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ADULTERATION AND ANALYSIS OF FOODS AND DRUGS

BIRMINGHAM METHODS AND ANALYSES OF SAMPLES
REVIEW OF BRITISH PROSECUTIONS DURING HALF A CENTURY

By
J. F. LIVERSEEGE
Fellow of the Institute of Chemistry
Pharmaceutical Chemist
Formerly Public Analyst to the City of Birmingham

With a Foreword
By The Right Honourable
NEVILLE CHAMBERLAIN, M.P.
Formerly Minister of Health



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DEDICATED

**TO MY PAST COLLEAGUES
IN THE BIRMINGHAM CORPORATION SERVICE,
TO WHOSE CAPABLE AND CAREFUL WORK,
AS ANALYSTS, INSPECTORS AND CLERKS,
THIS BOOK OWES SO MUCH.**

**AND TO
THE MEMBERS OF THE
BIRMINGHAM PUBLIC HEALTH COMMITTEE**

FOREWORD

AMONG the innumerable functions of the Ministry of Health, not the least important is its guardianship of the purity of the various foods and drugs supplied to the public, and, whilst I was Minister, I spent a considerable amount of time and thought on efforts to make this part of the work more effective. I had already been familiar with the administration of the existing Acts in the provinces, for I had served as a member of the Health Committee of the Birmingham City Council, and, in that capacity, had watched the actual working of the tests applied by the City analyst to the articles concerned. The result of my observations there, supplemented by the wider experiences of the Ministry of Health, satisfy me that, whatever criticisms may be directed against "bureaucracy" or "grandmotherly legislation," the public health is materially safeguarded by the precautions taken by the Local Authorities. Moreover, the protection thus afforded is obtained at a cost which is trifling, working out in Birmingham, as may be learned from Mr. Liversseege's interesting book, at less than a penny a year for each inhabitant.

I imagine, however, that Mr. Liversseege's work is intended not so much for the general public as for those who are actually concerned with the administration of the Food and Drugs Act, with public analysts and sampling officers, and with solicitors involved in prosecutions under the Act.

For such as are engaged in these occupations, Mr. Liversseege here offers valuable, because eminently practical, advice and assistance. Long acquaintance with his work in Birmingham enables me to say that, while few public analysts have had a more extended experience, none can have brought to their task a more conscientious thoroughness and reliability or a greater sense of fairness to all concerned.

I am glad to find that he has devoted his leisure, since his retirement, to making available to others some of the fruits of his long and, in some ways, unique experience; and I feel sure that he will, by this publication, earn the gratitude of many who are carrying on the work to which he has given so many years of his life.

NEVILLE CHAMBERLAIN.

PREFACE

ON more than one occasion, my old eminent chief, Dr. Alfred Hill, who was for forty years the Birmingham Public Analyst, after looking at a new analytical work, said to me, "That man is a book-maker." The feeling of disappointment is not uncommon in such cases owing to the large proportion in some of them of previously published work.

In my younger days I had an ambition to be a "book-maker," and write a complete and comprehensive treatise on the analysis of all foods and drugs. Since my retirement I have attempted the humbler, but probably more useful, task of giving an account of the analytical methods, research and memoranda, which have been found useful during my forty-three years' experience in the Birmingham Municipal Laboratory. No attempt has been made to duplicate information given in other works, but many references (more than 1,100) have been given to original papers, particularly those in the *Analyst* and Government Reports. For convenience of reference, general methods of analysis have been grouped together.

The records, which extend over half a century and cover 100,000 commercial samples of foods and drugs, have been generously placed at my disposal by the Birmingham Public Health Committee. From this abundant material analytical constants have been compiled, and in many cases percentage ranges of composition have been calculated—there are more than 200 tables. These should be of value to public analysts in performing their statutory duty of fixing limits and standards of composition. A recent judicial dictum, *Davis v. Blackman* (1924), has emphasised the much greater importance of evidence on minima than on averages.

As the main object of this book is "Adulteration," the dietetic value of foods is not discussed, and, as a rule, foods and drugs for which no prosecutions have been recorded are not included.

Particulars have been given as to the nature and proportion of adulterations detected in Birmingham, and the official figures of adulteration in England and Wales for fifty years have been summarised. These figures will be of use to sampling officers for indicating the relative liability of various articles to adulteration.

After a wide review of thousands of adulteration prosecutions recorded in trade papers, the writer has no doubt that many cases have been dismissed owing to the prosecution failing to produce

suitable evidence, and, on the other hand, a few vendors have been fined owing to their imperfect defence.

Many defendants have escaped conviction for adulteration owing to the certificates of public analysts not being in proper legal form. The writer has approached this question from an analytical point of view, and has discussed, with actual examples, some practical points not dealt with in legal text-books. He makes no claim to legal training other than the study of appeal cases, and attendance during thirty years at adulteration cases heard at the Birmingham Law Courts, during which time more than £5,000 has been paid in fines on his certificates. On such legal points as that of warranty, he has nothing to say. He hopes his work will help to prevent the waste of public money by preventing faulty certificates being given. Some observations on "Evidence" are also included.

The abstracts of about 1,400 prosecutions should be of use to solicitors engaged in adulteration cases, and also to analysts. They have been selected to show the variation in adulteration of a particular food or drug, and in many cases the arguments which led to the conviction or acquittal of a defendant have been given. Classification, according to the exact name of the article asked for, is attempted, but reports are sometimes indefinite. The reports of some old cases are of interest as showing when a particular form of adulteration was fashionable, and the improvement which has taken place. There is also the possibility of old adulterations being practised again. In Birmingham, after a prosecution for arrowroot, a large number of samples during a period of twenty-five years showed no sign of adulteration, and then there was a small epidemic of it.

In most cases it may be assumed that a fine was associated with the costs of prosecution, and at times they were much greater than the fine. Penalties also depend on the financial position of the vendor. The references, being usually given by date, will enable parallel reports in other trade papers to be found.

Particular attention has been given to accounts of prosecutions which led to appeal cases; the references to the latter are usually to non-legal journals; further references can readily be found in law books.

The methods of administration in Birmingham and particulars of its cost are given, and also examples of the diminution of adulteration. Methods of keeping records and forms are also included, and an account of "Sampling," with its methods and pitfalls. It is hoped that this study of the administration of the Adulteration Act by a municipality may be found of interest and suggestion to those engaged in the work in other districts.

Some notes, chiefly quantitative, have been given on microscopy, particularly of the starches, but for micro structures reference must

be made to special works containing illustrations, or to the British Pharmacopœia. The analytical methods of the latter are rarely included, particularly as it is in process of revision. Notes have been given from the Pharmacopœia Commission Reports of Sub-Committees. For the information of those who are not analysts, sufficient reference is made to the Pharmacopœia to render prosecutions intelligible.

The author hopes that the chapter on "Methods of Calculation" may be found suggestive, and that novelties in the factors given in the appendix may be approved.

Certain abbreviations (see p. xv) used by the writer for many years are systematically used, both for definiteness and economy of space, but they should cause the reader no difficulty.

An Index to Appeal Cases has been provided, and included in the detailed General Index are references by which the use, or rather abuse, of preservatives and adulterants can be traced.

The author wishes to express his very cordial thanks to many friends who have read and criticised parts of the MSS., and particularly to his successor, Mr. H. H. Bagnall, B.Sc., F.I.C., for checking the calculation of the factors, for reading the proofs and for giving facilities for including Birmingham results obtained 1928-31. I am also indebted to Mr. Joseph Marshall, the chief Birmingham sampling officer, for help derived from his long experience.

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ABBREVIATIONS

Analyst	Abstracts in the <i>Analyst</i> .
B.F.J.	<i>British Food Journal</i> (monthly).
B.P.	British Pharmacopœia.
B.P. Codex.	British Pharmaceutical Codex.
B.P. Conf.	British Pharmaceutical Conference papers, in Year Book of Pharmacy.
B-R. 25°	Butyro-refractometer readings at 25° C.
C. & D.	<i>Chemist and Druggist</i> (weekly).
E	Solutions approximately normal (see p. 552).
F. & S.	<i>Food, Drugs and Drink</i> , 1892-3 ; <i>Food and Sanitation</i> , 1893-9 (weekly).
gm.	Gramme.
J.C.S.	<i>Journal of the Chemical Society</i> .
J.S.C.I.	<i>Journal of the Society of Chemical Industry</i> .
K	Copper reducing power of a sugar, expressed as dextrose.
L.E.	Microscopical lycopodium equivalent (see p. 119).
L.G.B.	Local Government Board, which preceded the Ministry of Health.
μ	Micron, 0.001 mm.
ml.	Millilitre.
N.v/v	Millilitres of normal solution per 100 millilitres (see p. 112).
N.v/w	Millilitres of normal solution per 100 grammes (see p. 112).
P.J.	<i>Pharmaceutical Journal</i> (weekly).
Q.J.P.	<i>Quarterly Journal of Pharmacy</i> .
R.I. 25°	Refractive index at 25° C.
S.P.A.	Original papers and notes (not abstracts) in the <i>Analyst</i> .
v/v	Percentages by volume.
w/v	Parts by weight per 100 volumes ; grammes per 100 millilitres, etc. (see p. 112).
w/w	Percentage by weight in a mixture of liquids.
Y.B.P.	Year Book of Pharmacy abstracts .

ADULTERATION OF FOOD AND DRUGS

CHAPTER I

ADMINISTRATION. (i) GENERAL

Prosecution or caution? Cost of prosecutions. Effect of fines. Statistics. Comparison of various districts. Cost in Birmingham.

THE two classes of officers mentioned in the Adulteration Act are Public Analysts and Sampling Officers, and there should be close co-operation and, if possible, personal contact between them, to ensure the best administration of the Act.

The Public Analyst should be aware of current adulterations and able to advise which samples should be purchased, exactly how they should be asked for, how much should be bought, and if any special precautions are necessary in sampling. His analyses should indicate when formal samples should follow informal ones.

On the other hand, the Sampling Officer can inform the Public Analyst of any special circumstances occurring during the purchase of an article, particularly if any declaration about it has been made by the vendor. Many records of prosecutions suggest that if the Public Analyst had been given such particulars, unsuccessful prosecutions would not have been instituted.

There is no uniformity in the committee entrusted by an Authority with the administration of the Act. In the majority of districts it is the Public Health (or Sanitary) Committee, but in other places the Weights and Measures, Food and Drugs and Agricultural, Watch (or Police), or General Purposes Committee, that is responsible. Any discussion in committee should be on an adulterated *sample*, bearing a defining number, and not on a named *person*. A committee is not a judicial body. With regard to the choice of articles for analysis, the Public Analyst is very properly consulted in many districts, but in others the selection is made by the Medical Officer of Health, the Sampling Officer, the Chief Constable or the responsible committee itself. Obviously some of these officers cannot be expected to have expert knowledge of current adulteration and the possibilities of successful analysis of particular articles.

In large districts the employment of one or more whole-time sampling officers is the best plan. Samples are taken by sanitary

inspectors, weights and measures inspectors, and by the police ; but vendors should not be able to prophesy a visit from an officer for *sampling*, by his periodical visits for other work.

Adulteration certificates should be handed, or sent, to the Sampling Officer, and according to the Act, prosecutions may be instituted by him without the knowledge or consent of any official or committee (*Conner v. Buller*, 1902, Ireland) ; occasions may arise in which he must act on his own responsibility, but as a rule, he has to obtain the sanction of the Chairman of his committee, the Medical Officer of Health, or the Public Analyst, and the summons and certificate should be approved by the Town, or County, Clerk to be in legal form before being taken out.

The Sampling Officer should prepare a report on the purchase, including relevant facts that can be given in evidence. One copy of it should be attached to the certificate, and another given to the Public Analyst. Examples are given in the Appendix (p. 565).

Sometimes prosecutions are instituted of which the Public Analyst disapproves. There are samples which cannot be reported genuine, but for which prosecutions are inadvisable for various reasons. My practice in such cases was to report the sample adulterated, but not to fill up the legal certificate, upon which alone a prosecution can be based. If the Public Analyst is not prepared to go into the witness-box and support his certificate, he had better not give it in the legal form.

The question of informal sampling is considered in a later chapter (p. 12), but the undesirability of such samples being submitted to persons who are not public analysts for subjection to "rough sorting methods" may be here indicated. If samples are not properly examined it is a waste of money to purchase them. Another consideration is that informal samples, which are usually available in larger quantities than formal samples, can, when adulterated, be thoroughly examined, and the results obtained simplify the analysis of the often limited quantity of the formal sample.

A medical opinion on a case of adulteration is very rarely required. Many adulterations affect a consumer's pocket and not his health ; in most other cases, that a preservative in a particular quantity is injurious, or not, is settled by the figures given in the Preservative Regulations.

The Public Analyst is a Statutory Officer who is legally required to be highly and specially trained for his work, and the best arrangement is that he should be responsible for the administration of the Act, in consultation with the Chairman of his committee when necessary ; this is the method of the Warwickshire County Council.

Letters to caution offenders are best sent by the Public Analyst. The replies to them are at times highly technical, and a Medical Officer of Health who sends such letters may become involved in a

correspondence which he cannot continue without considerable help from the Public Analyst, and which, when half finished, he may even pass over to that officer.

In small districts, and when the Public Analyst's laboratory is not in or near his district, the administration of the Act is usually more or less in the hands of the Medical Officer of Health.

A paper by Hopkins on the relations between the analyst and the medical man was followed by a discussion (*S.P.A.*, 1906, **31**, 385).

PROSECUTION OR CAUTION ? The object of the Adulteration Act is to prevent substitutions which may be fraudulent, or injurious to health; and to prevent illegitimate competition being carried on. The problem of administration is to effectively stop such substitutions. Its success cannot be measured by the number of prosecutions nor by the amount of the fines obtained. Prevention being the object, prosecutions are often clumsy, ineffective, and, at times, an unjust method of doing it. Undoubtedly prosecutions have their place, but as the result of consideration and not of mere routine.

After a very expensive prosecution (not in Birmingham), the defendants expressed their willingness to label the article suitably, and said they had been prepared to do so from the first. A letter of caution in relation to new forms of adulteration will enable the Authority to obtain the point of view of the vendor and will often avoid expensive litigation.

It sometimes happens that several samples of milk bought from shops on one day are similarly low in fat, and part of milk supplied to the shops by one wholesale dealer. Prosecution of such innocent retailers appears to be unjust, though technical offences have been committed. Farmers who promise to eliminate unsatisfactory cows may fairly be given a further chance.

A vendor of an adulterated mixed spice and his wholesale dealer each had a warranty, and so, more than two prosecutions would have been necessary to obtain a conviction, but a letter of caution procured the removal of the defective article from sale. In another case a package of adulterated ginger was destroyed in the presence of the Sampling Officer. In 1913, samples of pearl barley bought in Birmingham had been faced with small quantities of talc, but a letter to the London firm concerned stopped the adulteration very near its source.

Another method which has been successful in Birmingham is to call the attention of the local Pharmaceutical, or Grocers', Association to the offending article. Sympathetic co-operation by the officials of the Association has followed, and circulars been sent by them to the members.

A lecture to the Association by the Public Analyst, and inquiries

addressed to manufacturers or letters to the trade Press have also been found useful.

Prosecutions for mistakes are usually unadvisable; the writer received a sample of "chicory" which was actually linseed meal. The article was accepted as "linseed meal" and passed as genuine. Elsewhere there was a prosecution for pea meal, accidentally sold as ginger.

In such cases as those given above, cautions have been more useful than prosecutions, in that, while the substitutions have been stopped, traders have appreciated that the action of the Authority has been for the protection of the public without the "persecution" of retail dealers. Occasionally a prosecution, without expecting a fine, may be the best way of obtaining publicity in respect to an adulteration. Also, it may be necessary to prosecute a retailer to ensure his evidence when a wholesale dealer is being prosecuted, and subsequently withdrawing the case against the retailer. Sometimes adulteration is done by a servant for his own profit. Of 292 Salford adulteration offences, 154 were traced to the producer or wholesaler, and 49 to the vendor (Report, 1930).

Before a vendor is cautioned for an informal sample, it may be advisable to obtain a second sample. This, if adulterated, will prove that there has been no mistake, that the adulteration was not accidental, nor due to natural variation, as may occur in jam. Informal samples not being divided, the vendor may have no check.

COST OF PROSECUTIONS. With new forms of adulteration the question may arise as to the justification of a Local Authority risking large sums of money in prosecutions which are really national problems. In 1900, Birmingham retailers were prosecuted for selling dyed sugar (yellow crystals) as Demerara sugar. The Corporation produced evidence from sugar brokers, manufacturers and analysts that the articles were distinct and sold wholesale at different prices. For the defence, several prominent retail grocers gave evidence that no distinction was made in the *retail* trade, and the cases were dismissed. This decision was unfortunate; it cost the Corporation about £135, though convictions were subsequently obtained in other places for similar offences; further, the decision made the purchase of other samples of Demerara sugar in Birmingham useless.

In 1920 the undeclared addition of saccharin to custard powder was referred to the Ministry of Food. The Government inquiries into preservatives in foods, and as to the nature of whisky, arose from very expensive local prosecutions.

A practical question of the day is "Should a vendor who sells dilute acetic acid as 'vinegar' be prosecuted?" The writer would not advise an Authority to do so, not because he has the slightest doubt that such a substitution is adulteration (see below, p. 389), but because it would probably lead to an unjustifiable expenditure

of public money. The vendors as a rule are small shopkeepers, who would probably be defended by expensive specialists, provided by the manufacturers, and if the case was lost the Authority might have to pay heavy costs. On the other hand, if the vendor was fined he would not be in a financial position to pay the costs of the experts called by the Authority, and so there would be loss to the Authority, whatever the decision of the magistrates. In the recent prosecution for table vinegar the magistrates refused to convict till directed to do so by the High Court (*Preston v. Jackson*, 1928), and though the vendor was fined £2, the County Council had to pay £60 costs, as the vendor could not pay them.

Until the uncertainty is resolved nationally by a Vinegar Act, or in some other way, it appears to the writer not to be advisable to prosecute for dilute acetic acid unless it is sold as malt or table vinegar.

EFFECT OF FINES. The only way to stop fraudulent adulteration, such as the addition of water to milk, is by prosecutions and by support by magistrates inflicting fines sufficiently substantial to make the fraud unprofitable. In 1874, samples of milk bought in Birmingham from four vendors on one day contained 29–60 % of water; each was fined 2s. 6d. ! It was not until 1881 that a farmer was fined as much as £10. In 1910–13 the average Birmingham fine per milk vendor was £3 17s., and the maximum was £25. In 1928–9 the average increased to £26, and the maximum fine per vendor to £100. When milk is sent to a city in quantities the fraudulent watering of milk is so profitable that the deterrent effect of even large fines may not last long (cp. p. 211).

Owing to the high price of spirits it is profitable to add even a small proportion of extra water. In the decade beginning 1909, Birmingham vendors of adulterated spirits paid £54 in fines, and the adulteration decreased from 15 % to 5·5 % in the next decade. In the early days the average amount of excess water was 16·5 %, in the last decade it fell to 4 %.

FINES IN BIRMINGHAM FOR ADULTERATION, 1873–1930.

	1873—	1897—	1889—	1899—	1909—	1919—	1929–30
VENDORS FINED .	80	228	589	410	250	115	29
PER VENDOR FINED							
Average fine .	18s.	£1 3s.	£1 18s.	£3 2s.	£7 2s.	£11 12s.	£12 3s.
Maximum fine .	£5	£10	£20	£100	£160	£80	£100

These figures should be compared with the falls in the percentages of adulteration given in the table below (p. 6). The decrease in the number of vendors fined and the increase in the average fine per vendor fined are noteworthy, particularly as there has been a great increase in the number of samples taken. The amount of the fines inflicted for adulteration, together with the fines for labelling offences and the costs paid by the vendors, amounted during the fifty-eight

years to over £8,100. It is impossible to estimate the financial saving the citizens of Birmingham have obtained by the suppression by these proceedings of various forms of adulteration.

NUMBER OF SAMPLES. In 1880 the L.G.B. considered that at least 100 samples should be examined for each 100,000 of the population. The Report of the Food Products Adulteration Committee, 1896, stated that this proportion was insufficient, and that "a local authority would do well to increase the number of samples taken from time to time until the number of adulterated samples found in those taken falls below the proportion which may be regarded as not unsatisfactory."

Some Birmingham figures for milk will illustrate this :—

MILK SAMPLING AND ADULTERATION

Period	1891-5	1896-1903	1904-9
Samples per 100,000 persons	67	87	147
Adulteration, %	17.4	14.6	7.3

If the number of samples taken be sufficient to impress fraudulent vendors with the fear that their fraud may be detected an increase in the number of samples would mean an addition of a proportional number of genuine samples, and therefore, a fall in the percentage of adulteration ; this is what happened in the third period, but the increase of samples in the second period caused a much smaller improvement.

STATISTICS. An increase in the percentage of adulteration may not be due to increased adulteration but to an increase in the proportionate number of samples particularly liable to adulteration, such as milk. Comparison of different districts, or the same district at different periods, may be misleading from this cause. This may be corrected by calculating to standard sampling (S.S. %) in which the adulteration figure is the sum of the number of adulterated samples found in fifty-two samples of milk, etc. (see p. 115), being the national proportion of samples in the classes.

PERCENTAGES OF ADULTERATION WITH STANDARD SAMPLING

PERIOD.	ENGLAND AND WALES.		LONDON.		BIRMINGHAM.		Average Samples per 100,000 Persons.
	%.	S.S. %.	%.	S.S. %.	%.*	S.S. %.*	
1873—					36.8	40.6	30
1879—	13.7	13.8	13.4	15.5	14.3	20.6	127
1889—	10.5	9.9	13.7	13.9	12.0	12.7	216
1899—	8.6	8.6	10.8	10.6	8.1	7.9	276
1909—	8.2	8.3	8.7	8.7	6.8	7.1	438
1919—	6.3	6.3	4.3	4.6	4.6	4.6	481
1929	5.4	5.4	3.7	3.8	5.5	5.5	495
1930	4.8	4.8	2.8	2.9	3.7	3.7	500

* Adulteration due to preservatives only is not included ; in 1929 the proportion was 0.2 %, and in 1930, 0.6 %.

A comparison of the two sets of figures indicates that the maximum difference for England and Wales was 0·6 %, and, therefore, that the proportions of the different classes of samples have shown little variation. The London figures vary a little more, but in one period the difference *due to varying the proportion of different samples* in Birmingham was over 6 %.

The figures for England and Wales show a steady improvement from 13·8 % to 4·8 %; the Metropolitan Boroughs, referred to as "London," have had a greater fall, from 15·5 % to 2·9 %. Birmingham, for which a longer period is available, showed a decrease in adulteration from 40·6 % to 3·7 %, with an increase to 5·5 % in 1929. This increase was largely due to systematic milk adulteration by a few farmers. No less than 62 adulterated samples were taken from five farmers. Had the sampling of these farmers ceased when a few samples had been taken the percentage of adulteration would have been considerably lower. The writer has used "comparative adulteration figures" (see p. 114) for diminishing this kind of error. The comparative adulteration figure for 1909-18 was 3·4, that for 1919-28, 2·4, and 1929 only gave a slight increase to 2·6. Adulteration with preservatives only is not included in any of these Birmingham figures. The table also shows the large increase in the number of samples taken in Birmingham, it being from 30 to 500 for each 100,000 of the population.

COMPARISON OF VARIOUS DISTRICTS. The following table has been calculated from figures given in the Reports of the Ministry of Health, and represents the averages of the five years 1925-9, of districts in which at least 5,000 samples were taken in the period. The populations used in those Reports are those of the census, without correction, except for alteration of boundaries. For this reason the number of samples per 100,000 persons for Birmingham is considerably larger than in the previous table. The amount of error introduced by using the census figures will vary with the rate of increase of population in the particular district.

The counties and the boroughs are each arranged in order of the amount of adulteration as shown by standard sampling. In the counties there is a distinct relation between the number of samples taken and adulteration. In the four best counties the average number of samples taken was 378, in the next four 253, and in the worst four 206 samples per 100,000 persons. In the boroughs the relation is much less obvious. For a similar comparison for 1893-7, see a paper by the writer (*B.F.J.*, 1899, 101).

When the proportion of milk samples is near the standard of 52 % of the total samples, the correction introduced by calculation to standard is small (0·0·1 %). In extreme cases there is a considerable difference; Staffordshire, where 77 % of the samples were milk, was 2·2 % higher than standard, and Essex, milk 36 %,

ADULTERATION IN VARIOUS DISTRICTS, 1925-9

DISTRICT.	ADULTERATION %.			STANDARD SAMPLING.	TOTAL SAMPLES.	
	Milk.	Not Milk.	Total.	Total Adulteration.	Milk Samples %.	Per 100,000 Persons.
<i>County :</i>						
Kent	4.2	0.6	2.6	2.4	57	459
Monmouth	4.6	0.8	3.4	2.8	69	284
Somerset	5.9	1.3	3.6	3.7	50	284
Essex	5.6	1.4	2.9	3.8	36	487
Glamorgan	7.0	0.9	4.9	4.0	65	220
Lancaster	5.9	2.4	4.4	4.3	58	301
Chester	6.5	2.6	4.5	4.6	49	233
York (West Riding).	6.5	2.6	5.0	4.6	61	260
Stafford	10.8	3.0	9.1	6.9	77	211
Surrey	7.8	8.9	8.1	8.4	75	311
Middlesex	10.5	14.3	11.9	12.5	62	119
Durham	20.4	5.6	12.0	13.2	43	183
<i>Borough :</i>						
Derby	5.2	0.8	2.4	3.1	35	400
West Ham	3.3	3.3	3.3	3.7	42	439
Bradford	4.4	4.1	4.3	3.9	67	355
Portsmouth	5.7	3.0	4.3	4.2	48	501
Birmingham	5.9	2.4	4.3	4.3	54	523
Liverpool	7.5	3.1	5.4	5.3	53	865
Salford	3.1	8.9	4.8	5.4	71	619
Bristol	8.0	2.4	5.4	5.5	54	370
Hull	6.5	5.2	5.9	5.9	50	411
Manchester	11.2	1.5	5.2	6.4	38	393
Leeds	10.8	5.9	9.5	8.5	74	304
Sheffield	9.8	8.7	9.5	9.9	75	224

was 0.9 % lower than with standard sampling. The districts would be in a different order if arranged according to the uncorrected percentage of adulteration.

The differences in milk adulteration are remarkable. West Ham, with 3.3 %, and Kent, with 4.2 %, are districts where many samples are taken. On the other hand, Durham has 20.4 % of milk adulteration, with a small proportion of sampling ; the high figure may be due to selection of suspected persons for sampling.

The percentage of adulteration in samples other than milk is equally remarkable. In Kent it was only 0.6 %, and in Middlesex 14.3 %, Surrey and Salford were also high with 8.9 %.

Some examples may be given of the proportions of standard sampling due to articles other than milk. For *butter* the highest amounts were 0.5 in Hull and 0.2 in Monmouth. For *spirits*, West Ham and Middlesex were each 0.5, and Durham 0.4. For *drugs*, Middlesex was 1.1 and Surrey 0.8. Some districts had no adulterated samples under each of these headings. For articles other than milk, butter, spirits and drugs, Middlesex had 5.1, Sheffield 4.2, and Surrey 3.2. The figures given are not percentages, but the amounts that each article contributed to the total standard sampling.

In the compilation of these figures large districts have been taken, and a five-years' period, so as to avoid errors due to small numbers, but it is very difficult to believe that adulteration in different parts of the country is so widely different as these figures indicate. A most interesting, but impossible, experiment would be to exchange the inspectors and analysts between a good district and a bad one, and note the alteration in the figures.

From his study of the question the writer has come to the conclusion that in the present lack of uniformity in conditions of sampling, any comparison of adulteration figures for different districts is probably misleading unless confined to very general statements.

COST OF ADMINISTRATION. The Birmingham City Analyst's staff consists of three qualified assistants, a clerk and a laboratory attendant. The writer has the opinion that a public analyst's laboratory is not a suitable place for training boys who "have done well in chemistry" at school, and has been supported in it by his committee. With slight exceptions in war-time all assistants appointed have had at least the B.Sc. degree. The work is too responsible to be entrusted to partly trained students, and they may be a positive danger if careless in their use of stock volumetric solutions. The samples are bought by three sampling officers, one of whom is partly engaged in taking bacteriological milk samples. An agent and a clerk are also engaged in the work.

The following table gives particulars of expenses for which there were definite payments. The cost of apparatus was a minimum, there being no special purchase during the period. The building occupied being shared by another corporation department, and rented from a third department, figures for rent, etc., cannot be usefully given.

AVERAGE ANNUAL COST IN BIRMINGHAM, 1923-7

CITY ANALYST'S LABORATORY		£
Salaries and wages		1,925
Apparatus and chemicals		43
Gas, water, electricity		37
Books, stationery, postage, telephone		38
Cost of cleaning		51
Accountant's charges		28
Total		<u>£2,122</u>
COST OF SAMPLING		
Wages		1,101
Purchase of samples, including tram fares		108
Bottles, paper bags, stationery, etc.		17
Total		<u>£1,226</u>

EXPENSES OF BUILDING, ETC.

Rent, rates, taxes, fire insurance.

Structural repairs, painting.

Interest on capital value of apparatus and fittings.

AVERAGE ANNUAL NUMBER OF SAMPLES ANALYSED IN BIRMINGHAM,
1923-7

Foods and drugs	4,781
Fertilisers and feeding-stuffs	15
Water and effluent	528
Various	103

Total	<u>5,427</u>
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The average amount received in fines was £103. In 1927 it was estimated that cost of the administration of the Adulteration Act, and the water and other analyses, was equivalent to one-sixth of a penny for each £1 of assessable value, or less than one penny per annum for each Birmingham inhabitant. This expense may be regarded as an insurance premium against adulteration. If the cost to Birmingham of foods and drinks covered by the Adulteration Act be roughly estimated to be 17 million pounds per year, the premium would amount to 5*d.* per £100 spent on these articles.

In 1899 Cribb and Moor made an estimate of cost of the Act and of various foods at that date in England and Wales (*B.F.J.*, 1899, 224).

CHAPTER II

ADMINISTRATION. (ii) SAMPLING

Informal sampling. Systematic sampling. Varied sampling. Amount to be bought. Definite and clear request. False labels. Conduct during purchase, observation. Containers, sealing. Note-books, wrappers. Refusal to sell, obstruction. Declaration, interval. Mixing before division. Division. Pre-packed articles. Place of delivery. Farmers' milk. Visits to farms. Persistent and varied sampling. Sending to consignors. Submitting to analyst. Reserve samples. Co-operation with neighbouring authorities. Bribery. Notification to vendors that a sample is genuine ?

To buy samples of foods and drugs is quite easy, but it may be little more than a waste of time and of public money, and with careless and unintelligent sampling the whole intention of the Adulteration Act for the protection of the public may be frustrated. To buy suitable articles in a correct manner from suitable vendors, particularly fraudulent ones, requires thought, and at times skilful detective work.

One fears that such absurdities as informing vendors *before* purchase, as "prosecution proceedings did not promote amicable relations with the police," or purchases by policemen in uniform (L.G.B. Report, 1911), are not yet obsolete. In 1924 in one county it was a standing joke that as soon as one shop was visited neighbouring tradesmen were informed by telephone or otherwise to be prepared to sell genuine samples, and in 1930 milk vendors were told to drive past the police station at 9 o'clock the next morning to be sampled. One has also heard of samples being taken by Weights and Measures Inspectors, whose visit to the district had been publicly notified. "Surely in vain is the net spread in the sight of any bird" is an important proverb for sampling officers.

There are, however, less obvious ways of informing an intelligent vendor that an article is being bought for analysis. The use of an unfamiliar name such as "spirit of nitrous ether" instead of the common one "sweet nitre"; buying an unusual quantity, as the $\frac{3}{4}$ lb. samples of lard, etc., recommended by the L.G.B. Circular (Feb. 26th, 1894); or the purchase of groceries by a *man* in the middle of the morning.

The article asked for should be suitable to the shop visited. Common drugs may be, and should be, bought at small hucksters' shops, and inferior, or adulterated, groceries are more likely to be bought there than at better-class shops. Drysalts and oilmen also should not be neglected.

INFORMAL SAMPLING

In recent years there has been a great increase in "informal sampling," and its recommendation by the Board of Agriculture and Fisheries Report, 1905, is worth reproducing :—

"It is the practice of certain Local Authorities to cause a number of samples to be taken without any of the formalities required by the Acts. Women and children are sent to purchase such samples. The object of taking samples in this way is to discover which traders, if any, are guilty of fraudulent practices. When one of these samples is found to be adulterated, further samples are taken of the same article from the same shop with the usual formalities and with a view to the institution of legal proceedings.

"This system has several advantages. It affords an effective means of testing the purity of the food supplied by the traders to their ordinary customers, as the buyers, not being burdened with unfamiliar responsibilities, make the purchases in a natural manner, whereas the agents employed by inspectors who are taking samples under the Acts frequently put the trader on his guard by their nervous and self-conscious behaviour.

"The system of informal purchases increases the chances of detecting habitual fraud, and at the same time diminishes the risk of the conviction of innocent persons who may contravene the Act by accident. It also saves unnecessary offence to honest shopkeepers whose chief objection to the taking of samples is that the inspector takes up their time and counter space for the division of samples and the ~~stealing~~ of packets, while his action excites curiosity on the part of the customers."

The following remarks on the improvement effected by the informal sampling of butter are from my Report for 1909 :—

"In June, 1905, a change was made in the method of sampling, and since then a large number of samples of butter have been bought informally. In 1906, the first complete year of informal sampling, the maximum adulteration of 14% was recorded. Since then the percentage of adulteration has fallen steadily, being 7, 6.5 and 6.2 in successive years. Under the old system whenever a sample was bought for analysis the vendor was informed and fraudulent vendors probably considered that another sample would not be bought for some time. Under the present system vendors do not know when samples are being taken for analysis, and so a series of informal samples can be taken at a shop where the fraudulent substitution of margarine for butter is suspected. Wrongdoers will often supply a stranger with the genuine article, but after several visits will think it safe to supply margarine. It appears probable that fraudulent vendors are finding out that even regular customers may be the agent of the inspector buying samples for analysis. I

think that this uncertainty has acted as a useful deterrent, and has helped to reduce the percentage of adulteration."

Another advantage of informal sampling is the saving of time. A formal sample must be taken by a sampling officer, and will probably require about a quarter of an hour, while an informal sample can be taken by an agent in a few minutes. In suitable premises an officer may wisely ask a vendor if he would prefer the division to be made behind a screen out of the view of customers. Is it necessary to add that informal samples which are found to be adulterated should be followed up in one way or other ?

SYSTEMATIC SAMPLING

In one place a year's samples were bought in *one day* and for the rest of the year adulterators were perfectly safe from interference. It is desirable that lists should be made of streets in the district in which there are shops, and of railway stations and wholesale depots to which churns of milk are consigned by rail or road, as well as arterial roads used by milk carts, so that in time all vendors shall be sampled. A sampling officer's hours should *not* be regular ; samples of milk should be taken early in the morning and late at night, on Sundays as well as weekdays, so that there is no "close time" for the adulterator. In 1905, 9 % of the Birmingham weekday samples were adulterated, but 24 % of the Sunday samples. Action was taken and 28 Sunday samples taken in 1912 were all genuine. The Sunday Observance Act, 1667, does not prevent a prosecution for a sample of adulterated milk sold on a Sunday (*Elder v. Kelly*, 1919).

VARIED SAMPLING. The articles sampled should be varied, with the exception of milk, which is always open to suspicion ; any fashion in adulteration should be followed, and if fresh articles are being tested it is an advantage to the analyst to have several samples submitted about the same time so that comparisons can be made. It may be advisable to ask him if an analysis of the particular article can be made. The percentages of adulteration given later under the various articles are an indication of their liability to adulteration. If a particular article is suspected it may be advisable to buy other common articles at the same time so that it may appear an ordinary purchase ; if the articles are associated, such as carbonate of soda and tartaric acid, so much the better. Proprietary medicines are outside the Act ; there is no standard, except the unknown one of the maker, and even that is often varied.

THE AMOUNT of an article purchased should, if possible, be that usually made by people of the class living in the district in which the shop is situated. It must be sufficient for an analysis to be made (*Lowery v. Hallard*, 1906), and that will depend on whether the

analyst receives the whole of it (informal sample) or only one-third (formal sample). In cases where the choice is between a barely sufficient sample or none at all, the way should be prepared by previously submitting an informal sample of the article. In other cases the cost limits the quantity to be bought, the $\frac{3}{4}$ pint of spirits advised by the L.G.B. (*op. cit.*) is now much too expensive, and a much smaller quantity suffices to detect excess water, the usual adulterant. Even the pint of milk, also suggested, may be too much for some small shops. In the Appendix a list of samples of food and drugs is given with the quantities usually bought for analysis in Birmingham, and the approximate cost.

There are devices for obtaining a sufficient quantity for analysis without suspicion. If the sale of margarine on bread is suspected, several people can go to a refreshment room and ask for an amount of "bread and butter" which would be unusual for one person. Similarly, glasses of spirits may be bought at a public-house for several men. Care must be taken, however, that one person gives the order and pays. Two men each bought a pint of beer, the *two purchases* were mixed and divided. The conviction for adulteration was, however, quashed on appeal (1920, *Grocer*, Jan. 24th; *B.F.J.*, 1920, 14). Sometimes a vendor has supplied an adulterated article when small quantities are asked for, and a genuine one for a larger sale. It may be advisable to ask for a small quantity, and when the officer sees from whence he is being served, to "change his mind" and double his order (*Payne v. Hack*, 1894).

DEFINITE AND CLEAR REQUEST. The name under which an article is asked for may require consideration. The order should be definite and clearly spoken so as to leave no doubt in the mind of even an inexperienced shop assistant. There have been remarkable cases of ignorance and stupidity, but these are very different from wilful fraud. Ambiguous orders, and traps for honest shopkeepers, are an abomination. The Adulteration Act is for the protection of the public, of which shopkeepers are an important part, and they are entitled to fair, if not generous, treatment.

There is no obligation on an officer to obtain "cases"; in fact, from a broad point of view, it is more satisfactory to buy a genuine sample than an adulterated one. The purchase of a genuine sample may be a tribute to the previous efficient work of the department.

A request must be made for a definite article. An agent was given the choice by the shopkeeper of two articles, and said, "I will take that." It happened to be adulterated, but no action could be taken for an article bought only as "that." In another case "1s. Salt" was held to be an insufficient description for butter (see also *Sandys v. Jackson*, 1905).

In ordinary cases the use of the word "pure" is unnecessary. In a yeast case, *Bell v. Mallison* (*B.F.J.*, 1904, 109), the Lord Chief

Justice said a vendor must supply the article he was asked for in a pure condition.

Some articles are sold of different qualities for commercial and internal use. A small quantity of "borax" bought either from a chemist or a druggist, might be for laundry use and therefore outside the Act, being neither a food nor drug. For "purified borax" there is a definite limit of purity (p. 100), or a question may be asked, "Is it right for mixing with honey?" to indicate it is for internal use. Pills and some other drugs are sold in different weights, and the demand should be definite, "four grains blue pills" for instance.

If an agent be sent for an uncommon drug it is well to send a written order for the quantity required, adding the letters "B.P." if advisable, about which the analyst should be consulted. For other articles a simple prescription may be necessary. I had reason to believe that some very inferior "infusion of senega" was on the market, and as that article is never asked for over the counter it was ordered in a prescription and prosecutions followed (cp. p. 529).

FALSE LABELS. There are articles for which no standard has been established, such as "Glycerine, honey and lemon." If, however, definite claims are made on the wrapper which are found by analysis to be incorrect, the vendor is liable to prosecution under sect. 30 (1) for giving a "false label." It is necessary, however, that the offence be "wilful." Great claims made for some articles in advertisements may not be put on the labels. In such cases the article may be brought under the Act by taking the advertisement to a shop and asking for the article advertised.

CONDUCT DURING PURCHASE, OBSERVATION. It is of the greatest importance that any reply of the vendor to the request for an article should be noted, either disclaiming any responsibility for its purity, or offering another in its place, or by giving a printed notice (*Kirk v. Roythorne*, 1924). Unless the vendor has been cautioned any committal statements should not be used in evidence.

A sampling officer should not be afraid of appearing a bit of an ignoramus. With informal samples in particular, debate with a vendor is very undesirable, because it will call attention to his person, and probably ensure recognition as a suspicious person at any subsequent visit. Recently a Birmingham officer asked for "calomel ointment." The vendor did not appear to know the article, and suggested that "calamine ointment" was wanted. The officer smilingly accepted the correction, bought the article, and submitted it for analysis as "calamine ointment." It turned out to be an unsatisfactory sample, and the vendor was cautioned.

An admission by a vendor that he has sold an adulterated article does not obviate the necessity for declaration and analysis (*Smart v. Watts*, 1895).

An officer should be observant. On one occasion "almond oil" was asked for, and the article sold was labelled as such. The officer noticed that the bottle from which it was taken had on it the word "persic" and though he did not know that the word indicated a substitute, reported the fact to me. His observation proved that the vendor had "wilfully" given a false label, and a penalty followed. A milk vendor nearly succeeded in supplying an inspector with genuine milk from a bottle while pretending to supply milk from a can, the latter being adulterated.

When milk is bought from a cart the vendor's name and address should be looked for on it (1915 Act, sect. 6), and also if there be any notice of dilution or abstraction on the serving can (*ibid.*, sect. 7). Statements as to churns being empty should be checked by actual inspection, or mixtures of milk and water may be missed. Sometimes all liquids on a cart should be sampled (*B.F.J.*, 1918, 3). Carrying water on a milk cart is very suspicious, but not an offence in England, though it is in New Zealand (*Analyst*, 1928, **53**, 593).

In cases where margarine is exposed for sale without being properly labelled, careful note should be made of the conditions. It must be exposed so as to attract the attention of customers, *Myers v. Gregory* (*B.F.J.*, 1906, 29); one notice is sufficient for six separate pounds which touch, *Parkinson v. M'Nair* (*B.F.J.*, 1905, 155). Sometimes sketches and measurements may be usefully made, or when a notice is given by a placard, the evidence of photography may be secured. On cross-examination the officer should show that his observation has been exact and accurate.

When spirits are purchased the officer should be prepared to give exact evidence as to the absence, or presence, at the time and place of purchase, of a dilution notice, its wording, its obviousness to a customer, the calling of his attention to it, and if that was done before the completion of the purchase. References to cases are given on p. 367.

Similarly, observation should be made if a preservative notice is in due form and "exhibited in a conspicuous place so as to be easily readable by the purchaser."

Observation of the presence of persons also may be necessary. It has happened that, at the retail sale of an article, a commercial traveller representing the wholesale dealer has been present, and has later been produced as a witness for the defence. Such evidence may be based more on suggestion than on memory, and may purport to support the evidence of a conversation which he could not possibly have heard.

CONTAINERS. SEALING. A circular of the Board of Agriculture (Dec. 28th, 1901) advised that bottles should be of the round "Winchester" shape, closed with new and closely-fitting corks, sealed so as to prevent any unauthorised removal of the

contents (cp. *B.F.J.*, 1931, 77), and of such a size that the sample of milk about fills them.

A Memorandum of the Ministry of Health (36/Foods, Jan., 1929) directs that samples of butter, dried milk and similar fatty substances shall not be wrapped in paper, but put in wide-mouthed bottles closed with screw-capped metal lids lined with cork, or in similar receptacles. The bottle should be placed in a sealed envelope, and both the bottle and the envelope should be numbered.

A poor cork was adversely criticised in a distilled water prosecution. Cases have been dismissed owing to unsatisfactory attempts to seal a metal cap securely to a glass jar (see artificial cream, p. 267).

The cleanness and dryness of sample bottles must be above suspicion; a wet bottle was urged as a defence in a camphorated oil case.* Difficulties have arisen owing to the imperfect closing of bottles containing spirits (p. 369) and sweet nitre (p. 493), and in one case the composition of a sample of sausage altered by evaporation, owing to inefficient wrapping (p. 294). In another case, the wrapping of a baking powder in paper was considered unsatisfactory.

With milk samples it is advisable not only to seal the cork of the bottle left with the vendor, but also to put a seal on the label (cp. *B.F.J.*, 1910, 14; 1929, 40). Cases have been known in which the label belonging to an adulterated sample has been transferred to a similar bottle containing a genuine sample.

It may be noted that the Act (sect. 18 (1)) allows the alternative "fastened up" to sealing.

NOTE-BOOKS, WRAPPERS. It is an advantage for samplers to have printed note-books, which include three detachable gummed, numbered labels for each sample (see pp. 29, 564). The note relating to a formal sample of milk from a shop should include the name of the wholesale dealer, and his hours of delivery, so that if it be adulterated no time is wasted before sampling him. A similar question should be put to a wholesaler about his farmer. Notes of a purchase should be made at the time, or as soon after as is possible. Such notes may be used in evidence.

With informal samples the article, with its wrappers or labels, should be shown to the analyst; with formal samples careful note should be made if any declaratory label was visible before the completion of the purchase, and if attention was called to it. Any metal capsule should be removed before sampling. In one case it was suggested that the lead present was due to the liquid running over the capsule during sampling.

In prosecutions any original bottle, container or wrapper should be taken to court to be produced if necessary. The importance of this in unlabelled margarine cases is obvious, and may be useful in

* See Addenda, p. 577.

other cases. The exhibition of a correct wrapper may help the magistrates. A defendant will also see he is having fair treatment and there is no hiding of facts. A sampling officer is not responsible for the legal aspect of a case, but unless he is an accurate observer of facts he fails in his duty to help both his Authority and the magistrates to do justice.

REFUSAL TO SELL, OBSTRUCTION. An officer has no powers of search, and if a vendor denies having an article the officer, as a rule, whatever his suspicions, can only say he "will call again" and try to get a sample by an agent.

If the article be "exposed for sale," refusal is an offence, but an officer should always carry with him a certified copy of his appointment to show, if his authority be challenged (*Payne v. Hack*, 1894). Money must be produced and offered to the vendor.

If the driver of a milk cart refuses to stop when asked to do so by a sampler it is necessary to be able to identify the driver if there be any prosecution for obstruction.

Refusal to sell milk exposed in a restaurant is an offence, even if the milk is only used for mixing with tea, *McNair v. Terroni* (1915; *B.F.J.*, 1914, 232), and a sampling officer is entitled to demand to be supplied in the same manner as the public, *Soutar v. Kerr* (Scotch) (*B.F.J.*, 1907, 45).

Occasionally refusal to sell is for a very good reason and may be followed by obstruction, as in the following case. A wholesale dealer refused to serve a Birmingham inspector with milk, and emptied the 2 gallons of milk in it on to the road. Another churn, which was said to be empty, contained 6 gallons of milk, and as the vendor was also emptying that churn into the road, the inspector managed to catch some in a jug. The vendor then struck him and spilled most of the milk; the part saved contained 18 % of excess water. For refusal to sell, obstruction, and assaulting the inspector, who was a police constable, the vendor was fined £15 5s.

A purchase should be kept out of a vendor's reach so that he cannot snatch it back. At Woolwich a publican was fined £10 for seizing and retaining a bottle containing gin which the sampler's agent had bought (*B.F.J.*, 1929, 114).

A case relating to another variety of obstruction was heard at a London (Old Street) police court. While a chemist was dispensing a test prescription the dispenser from another shop where a prescription had just been dispensed entered the shop and had a whispered conversation with the chemist, and warned him to be careful. The magistrate considered the dispenser had both obstructed and impeded, and ordered him to pay 5 guineas costs (*P.J.*, 1925, Jan. 3rd; *Analyst*, 1925, 50, 70).

DECLARATION, INTERVAL. "After the purchase has been completed" (sect. 18) the vendor must be told that the article is

to be "analysed by the public analyst" (*Barnes v. Chipp*, 1878), but if the declaration be made to an assistant it need not be repeated to the owner (*Davies v. Burrell*, 1912). With an agent the purchase will be complete on payment, but a sampling officer may declare the article immediately it is handed over without waiting till payment has been made (*Miles v. Melias, Ltd., B.F.J.*, 1930, 34; *Analyst*, 1930, 55, 326; see p. 171). When an agent has made a formal purchase it is desirable that she shall not go further than the shop door to signal to the officer, but an interval of two minutes is not fatal to the prosecution (*Somerset v. Miller*, 1890). The interval must not, however, be long enough to make it *possible* for the article to be changed. An agent should be asked by the officer in front of the vendor, "What did you ask for?" and "How much did you pay?"

The case *Cox v. Evans* (1917; *B.F.J.*, 1916, 429) confirmed a conviction for an adulterated milk, in which the churn on arrival at a railway station was taken in charge by the police for 20 minutes before sampling. "There must be an intervening period, which would vary according to the facts of the case." It is however better for the sampling officer to be present when the train arrives.

During a strong wind at Dundee the sampler, after corking the bottles, took them out of the vendor's sight and sealed them. A conviction followed (*B.F.J.*, 1931, 8).

MIXING BEFORE DIVISION. The question of mixing is of importance both to the vendor and to the analyst. To the vendor that the sample taken is a fair one, and to the analyst that the three parts of the sample shall be *identical* in composition. A prosecution may depend on the comparison of the work of three analysts, and without uniformity in the three parts agreement is impossible, and analysts may be discredited when accurate analyses have been made. Three parts of a sample of milk contained 2.28 %, 2.83 %, and 6.65 % of fat respectively (*B.F.J.*, 1926, 120).

A sample of "butter" was sent to a public analyst, and found to be chiefly margarine, while that sent to the Government analyst was butter; the two ends of the sample being different. Two parts of a sample of suet contained 22.2 % and 15.5 % of flour. Three parts of a sample of coffee contained 25 %, 5 % and 1 % of chicory respectively. These results show the necessity of mixing solids. Some jams require thorough mixing, and so does condensed milk (*Backe, S.P.A.*, 1911, 36, 138).

Churns of milk must be thoroughly mixed. Experiments made by the direction of the Board of Agriculture and Fisheries (Intelligence Division Report, 1910, p. 66) indicated that six rapid movements of a plunger from top to bottom, or pouring into an empty churn and back three times satisfactorily mixed the milk, but the use of a dipper for mixing was ineffective.

Bottled milk cannot be satisfactorily mixed by shaking ; the contents should be emptied and thoroughly mixed (Tocher, *Analyst*, 1927, **52**, 472). When milk is bought from a bowl in a shop the vendor should be told to mix the milk with the measure.

It is probably impossible to correctly sample milk which has been partly frozen. Vieth (*S.P.A.*, 1886, **11**, 69) examined two samples of partly frozen milk and found differences of 2·7 % and 3·2 % of solids-not-fat between the liquid part and the melted ice, with similar deficiencies of fat. Should such melted ice be sold by itself serious amounts of "added" water would be indicated. In one such case the vendor was fined £5 for each of two samples, it being held that the customer had not been supplied with what he had a right to expect (*B.F.J.*, 1924, 25).

In a case recorded by Estcourt (*S.P.A.*, 1879, **4**, 51), it was maintained that excess water in a sample of milk was only apparent, being really due to the sample being taken from the top of very cold, but not frozen, milk. An experiment in these conditions showed a slight deficiency in solids-not-fat due to cream having risen. Previous mixing of the milk would have prevented any such argument.

DIVISION. In most cases the division of the sample into three parts presents no difficulty ; they need not be equal, but each must be sufficient for analysis (*Lowery v. Hallard*, 1906). Equal division in cases where a small barrel is bought wholesale would be awkward. Further, exact division into equal parts is not always easy. I took three oval sample bottles of 6 oz. capacity, and divided 6 oz. of water between them so that the liquids in the three bottles were on a level, the contents measured as follows :—

Experiment I. Bottle 1 = 60 ml. Bottle 2 = 63 ml. Bottle 3 = 52 ml. Total 175 ml.

Experiment II. Bottle 1 = 58 ml. Bottle 2 = 65 ml. Bottle 3 = 52 ml. Total 175 ml.

The bottles were not selected, and after measuring the liquid it was found that the glass at the bottom of No. 3 was much thicker than the others. The maximum difference was about $\frac{1}{2}$ oz., or about 25% of the smaller. In the second experiment liquid was poured eight times, and the time taken was 50 seconds. The division was a copy of one actually done, but the bottles were undesirably large for the quantity of liquid.

In one case a sampler bought 1*d.* worth of milk, declared and divided it, then, thinking it did not look enough, bought another $\frac{1}{2}$ *d.* worth, and divided that among the bottles. The prosecution was dismissed ; nothing must be added to a sample.

When sampling liquids, care must be taken that the three bottles provided shall be big enough to hold the purchase, even allowing for overmeasure. Six quarts of beer were bought from a publican, and put into two half-gallon bottles, and two quart bottles. The

case failed, as division was into four parts, not three. In one case it was elicited in Court that an officer, after filling his bottles, drank the remainder of the milk ; again a division into four parts.

When a mixture prepared according to a prescription is sampled, the height of the liquid should be marked on the bottle before division. The original bottle is taken to the analyst, so that he can measure the total quantity, and also check the directions for taking the medicine.

When articles are composed of two dissimilar parts, like tins of preserved peas, inspectors have been known to throw away the liquid, and divide the peas only ; again a division into four parts. The peas and the liquid should each be divided as equally as possible. Potted meat with a layer of fat on the top should be treated similarly.

Also the sampling must be fair. A sample of currant cake was so divided that the part sent to the Government Analyst consisted only of outside parings and scrapings. He refused to accept it as a fair sample, and the case was dismissed.

It may be noted that the obligations to notify the vendor and divide the sample *only* apply when " the persons purchasing a sample of any article *with the intention of submitting it to analysis* " (sect. 18), and not when an article is bought for food purposes, the decision to have it analysed being made later.

PRE-PACKED ARTICLES. The division of such articles requires some discussion. From a small shop six 2*d.* bottles of camphorated oil were bought ; two unopened bottles were put in each of three bags and sealed. This proceeding was declared incorrect on appeal, *Mason v. Cowdary* (*P.J.*, 1900, June 9th ; *B.F.J.*, 1900, 162). Evidence was given that the vendor had bought the bottles of oil, but it did not show if from *one* chemist, or from two or more. An inspector bought four packets of cream of tartar, they were taken from one box, and were similar in appearance and label. The four packets were emptied, mixed, and divided into three parts. The Lord Chief Justice ruled that the division was satisfactory, but thought some question might arise in the case of mixture of articles coming from different people, *Smith v. Savage* (*Analyst*, 1905, **30**, 179 ; *B.F.J.*, 1905, 77). The principle underlying each decision is clear, the three parts of a sample must be identical. A prosecution at Woolwich was dismissed because two different makes of camphorated oil had been mixed. The special case of Seidlitz powders is considered later (p. 444), and also the precautions necessary in sampling sweet nitre (p. 492). Such articles as apples, sponge cakes, and cakes of beeswax, should each be divided into three parts, as well as sticks of chocolate cream (*B.F.J.*, 1903, 93).

In a recent case, two 6-oz. bottles of a preparation were bought, they were not mixed, but successively divided between the three

sample bottles. The two bottles bought were from one maker, but there was no evidence that they were made at the same time, and they may have been different in composition. To test the amount of inaccuracy of such a method of division, 6 oz. of water was divided into three 6-oz. dispensing bottles, as equally as was possible to the same level, the quantities were measured, the water returned to each bottle, and a second 6 oz. added in the same way, and the total volume measured. It was found that in six trials the relation of the first part to the whole varied from 50 to 56 %. These differences, determined in the laboratory, were not large, but sufficient, if the contents of the two bottles were not of the same composition, to prevent the contents of the three samples being identical. Divided in a shop, the differences would probably have been greater, and if with irregular sample bottles like the previous experiments, much worse.

PLACE OF DELIVERY. Samples of food, but unfortunately not of drugs, can be taken at the place of delivery "upon the request or with the consent of the purchaser" (sect. 16 (2)). This is a useful provision for cases in which it appears probable the retail vendor is being victimised by the wholesaler. Care should be taken that the order sent is in suitable terms, and that there is no declaration of admixture. For instance, "vinegar" might be ordered and the cask sent found to be labelled "artificial vinegar." The purchase of a formal adulterated sample from the retailer should always precede action under this section, and he should be served with a summons, not necessarily to obtain a penalty from him, but to secure his attendance at the prosecution of the wholesaler, at which he is a necessary witness (cp. p. 24).

Sect. 6 (1) of the Preservative Regulations, 1926, gives power to inspect premises in which articles to which they apply are prepared, packed, labelled or stored.

FARMERS' MILK. When samples are taken in course of delivery at a railway station, no declaration can be made. In Birmingham, consent has been given by wholesale milk dealers that sampling officers may take an *informal* sample from any churn coming in by train or road. The samples are brought at once for analysis, and if any sample be found to be deficient, a formal sample is taken the next day from a corresponding churn. The quantity of milk in each churn is observed, and its guarantee tally removed in exchange for one written for the purpose. As a rule, a preliminary report can be obtained before post time, and if the second day's sample is genuine, it is made informal, and no sample is sent to the farmer; otherwise, the sample is sent to him and a further sample taken on the third day. Frequently, when the milk is sent from a distance, the third day's consignment has been sent before the arrival of the post, and it may also be adulterated. The farmer

then knows that his milk is being tested, and the fourth-day samples often show a great improvement in quality and diminution in quantity. Usually on the morning of the fourth day he receives an invitation by post to have his milk sampled at the farm, with a prepaid telegraph form for reply. On receipt of an acceptance of the offer, sampling officers proceed to the farm, take samples and divide them in the usual way. With this procedure the milk of five successive days is frequently obtained, and any suggestion of any change in the quality and quantity of the milk is disproved. This proceeding may appear somewhat cumbrous, but it has been successful, and fines of £100 have on several occasions been obtained from fraudulent farmers, as well as a number of substantial fines.

In some districts sampling officers go direct to the farm and ask for permission to sample the cows' milk there, without any previous communication concerning a visit.

The identification of milk as "morning" or "evening" is often of importance, and the relative temperatures of two churns of one consignment may be helpful if taken.

In recent years there has been a great increase of road transport of milk, and some farmers have preferred it, thinking such milk was safe from sampling. Further, milk delivered at a depot is often quickly bulked in a pasteuriser with other milk, and adulteration is then covered. Systematic sampling at wholesale milk depots of milk on arrival by road has in Birmingham interfered with adulterators' plans.

In certain cases milk can, under the second schedule of the Act, be taken in course of transit.

When a consignment of milk consists of several churns, it is usual to take a sample, and if adulterated, issue a summons for each churn. The alternative of issuing one summons for a consignment of three churns and calculating the adulteration from the respective quantities and composition of the three churns, was considered satisfactory in *Wildridge v. Ashton (B.F.J., 1923, 105)*.

In a Scotch case one churn out of a consignment of three was adulterated, and the sheriff overruled the suggestion that the contents of the three churns should have been mixed and the adulteration given on the whole bulk. The sampling of each churn was satisfactory (*B.F.J., 1927, 107*).

It is not necessary to take milk as it is being poured, as "delivery" is not completed when all the milk has entered the purchaser's vessels, if the milk has not left the eye of the inspector (*Birch v. Eldred, B.F.J., 1925, 56*).

A case in which a sample of milk was taken from a customer a short time after she had shut the door was dismissed as not taken in course of delivery, *Helliwell v. Haskins (B.F.J., 1911, 90)*.

Sometimes there is difficulty in deciding which is "the place of delivery" when milk is sent by rail or road. The question as to who pays carriage is an important one, and a careful examination of the contract may be necessary. On this point a Gloucester farmer escaped, and boasted of his addition of water to milk : "I beat 'em. I put a drop of water in, it keeps the milk better. I am doing it now." The milk was then going to London.

VISITS TO FARMS. It must be remembered that samples at the farm, except in course of delivery, can only be taken by the consent of the farmer, and he should be warned that he need not say anything, and that any statements made may be repeated to the magistrates if there be a prosecution. A farmer will usually give particulars as to the breed, age, stage of lactation, and the time of the previous milking of the cows. A specimen report of a visit to a farm is given in the Appendix (p. 566).

The farmer must be asked to duplicate the conditions which existed when the suspected milk was taken from the cows. The time, the cows, and if possible the milkers, should be the same, or any difference be carefully noted. The number and condition of the cows milked, the yield, and the time of commencing and finishing milking should be recorded. Inquiry should be made if there are any sucking calves, which may sometimes be identified by a rope round the neck, or by the noise they make when separated from their mothers.

The cooler should be tested with the water turned on ; if a leak is discovered an attempt should be made to estimate the rate of flow, and the hole examined to see if it resulted from wear or intentional injury. The churn into which the milk is put must be *proved* to be empty. The milk should be sampled before cooling, and also taken after if the farmer desires it. A sample of water may also be taken to be tested for nitrates.

When fat has been found to be deficient, the question of the cows being milked is an important, and perhaps difficult, one. Elsdon has reported a case (Salford Report, 1923) in which three visits to a farm were necessary to obtain a fair sample of milk. On the first two occasions only about 25 gallons of milk were obtained, but when three milkers, unconnected with the farmer, milked the cows, 39 gallons were obtained. On the first occasion one of the farm servants managed to introduce 10 % of water ! If the cows are gone over a second time the distribution of the "strippings" between the churns must be noted.

Sometimes a separator, or the presence of cream, or butter in the dairy may be observed. The conditions in which milk is stored over night may also require attention.

The number of visitors must be adequate ; milk sheds are sometimes dark, and distant from the churn and cooler ; farmers

have attempted to put milk to water already in the churn, and all round vigilance is necessary (cp. *B.F.J.*, 1931, 88). In a recent case an inspector went and had his breakfast while the cows were being milked; 7 % of water was found!

One case was dismissed as there had been an interval of four days before appealing to the cows (*B.F.J.*, 1931, 99).

PERSISTENT AND VARIED SAMPLING. At times considerable patience and perseverance are necessary before an adulterator can be convicted, as in the following case. A sample bought from a shop on January 20th was adulterated with 20 % of water. The shopkeeper very unwisely told the wholesale dealer, and a sample bought at the shop two days later was genuine. Three later samples were genuine, or nearly so, but one bought at the shop on February 20th contained 11 % of water, and was coloured. Two samples taken from the wholesale dealer the next day were of similar composition, and he was fined £10.

Adulteration of foods delivered to order at private houses is very difficult to prevent, but some householders may permit a sampler to take samples at the time of delivery. An agent may be introduced as a new customer, and such a purchase will prevent the necessity of a householder's attendance at a prosecution.

With persistent adulterators, variations in the method of sampling may be necessary. In 1904 a Birmingham farmer was fined £25 for the retail sale of milk on two dates containing 9 %, 14 % and 20 % of added water. He is reported to have said that the retail sale of milk was "too risky," and in July, 1911, three samples of his milk taken in course of delivery to two wholesale dealers contained $5\frac{1}{2}$ %, 9 % and 22 % of added water. As only half of the road in which the delivery took place was in Birmingham, the inspector arranged for a cart to stop on the far side of the road to prevent the farmer delivering the milk out of the inspector's district. The farmer was fined £5. After this the farmer declined to deliver milk to anyone except at his farm, and in November, as adulteration still continued, arrangements were made for the retail dealer buying the milk to take a Sunday sample as a private purchaser, and submit it to me. This milk contained 8 per cent. of added water, and a fine of £20 followed. The farmer then left the district.

Under sect. 4 of the 1922 Act, the addition of water to milk is an offence, even if the mixture is above the limits of the Milk Regulations. Occasionally the addition may be seen, as when a milkman was observed from the bridge of a railway station to add $1\frac{1}{2}$ gallons of water to milk which had just come by train. On these occasions the water is usually stolen.

An unusual cause of deficiency in milk was indicated in a prosecution at a London (Tower Bridge) police court. Milk from an automatic machine was deficient in 12 % and 12·7 % of fat

respectively. The defendant, who blamed the machine, was fined £3 and 10 guineas costs (*B.J.F.*, 1929, 90).

SENDING TO CONSIGNOR. Part of a sample taken in course of delivery is required to be sent to the consignor by registered parcel post or otherwise, if his name and address appear on the can or package (sect. 18 (2).) In other cases the Birmingham practice has been to obtain his address from the consignee. A letter is enclosed in the parcel, informing him that a sample of his milk has been taken to be analysed by the Public Analyst, and giving particulars of the consignment and its place of delivery. On paying an extra 3d. at the Post Office for an "A.R." form, a sampling officer can receive by post a receipt for the parcel signed by the farmer or his agent, which can be produced at Court to prove that the consignor has received the sample.

SUBMITTING TO ANALYST. Samples should be submitted to the analyst as soon as possible after purchase, preferably on the same day if "liable to decomposition." With formal samples there must be no break in the evidence as to the transmission of the sample from purchaser to analyst. It is therefore desirable that the sampling officer shall *personally* submit the samples to the analyst, unless they are sent by registered parcel post. Anyone coming between should attend at Court to prove, if necessary, his share in the matter.

When a sample or samples are submitted, a sheet, giving the name of the officer, the date, and the name under which the article has been bought, should be supplied to the analyst with them. It is convenient for these lists to be on forms which can be signed by the analyst for the articles which are genuine; any adulterated being crossed out, and certified separately. Forms used in Birmingham for many years are given in the Appendix (p. 570). Red ink was used for informal and black for formal samples.

In certain cases, where the deficiency in quality, etc., cannot be determined by analysis, the article is not divided and submitted to the Public Analyst, but to experts with regard to the particular food. An example is given under "New-laid Eggs"; a meat expert would be required to prove if beef was "English," or a fish expert to prove if a fish complied with the description under which it was sold.

RESERVE SAMPLES. These should be kept locked up in a cool dark place and produced at the prosecution (*Scotch Appeal, Hutchinson v. Stevenson*, 1902). It may be necessary to prove the identity of the number on the label with that on the certificate.

In one instance the Government Chemist reported that a bottle containing whisky arrived half full and was imperfectly corked. In another case his report was that fermentation had partly forced out the cork of a sample of milk and that some of the contents had escaped, making analysis impossible. On appeal (*Suckling v. Parker, B.F.J.*, 1906, 70), the magistrates were instructed that if

the sample had been "sealed or fastened up in such a manner as its nature will permit," a conviction should follow in spite of the Government Chemist being unable to analyse it. In a sardine case, about a month after purchase the defendant's analysts were unable to obtain any oil for analysis from the sample, and at a later date the Government Analysts reported that the mouldy condition of the sample prevented an analysis. The Stipendiary convicted, being satisfied that there was no negligence or want of care on the part of the inspector. On appeal (*Winterbottom v. Allwood*, *B.F.J.*, 1914, 78, 99, 209), the conviction was confirmed.

Should a sample burst or be otherwise destroyed, the pieces or remainder of the sample should be produced in Court. A farmer was fined £20 at Liverpool in connection with a milk when the bottle containing the reserve sample had been broken (*B.F.J.*, 1902, 200).

Of the 6,496 samples reported to be adulterated in 1930, only twenty were referred to the Government Chemist.

CO-OPERATION WITH NEIGHBOURING AUTHORITIES. In some instances, co-operation between two neighbouring Authorities is very useful. In 1915 a farmer who was selling water as milk to a Birmingham wholesale dealer at the rate of about 73s. per week, was fined £30. He then arranged for a Birmingham wholesaler to fetch his milk from his farm, and continued the adulteration. As his farm was outside the city, information was given to the County Authorities. Three samples taken by them contained 8 %, $12\frac{1}{2}$ % and 24 % of water, and he was fined £102. Samples taken at the farm indicated that the natural milk was of good quality. One morning the inspector visited the farm, saw some of the cows milked (the farmer had started extra early), and noted the quantity to be $15\frac{1}{2}$ gallons. In the evening he returned to see the cows milked again; the churn of morning milk had not been fetched, and he, observing that the quantity was then 17 gallons, took a sample. This sample, which contained 24 % of added water, was one of those for which a fine was inflicted.

On another occasion, owing to a complaint, this Warwickshire inspector watched a Birmingham retailer, who had just fetched two churns of milk in his cart from a Warwickshire farm, and saw him add water and colouring matter to the contents of the churns. A Birmingham inspector took samples of the milk when it arrived within that area. Analysis indicated the presence of $2\frac{3}{4}$ gallons of added water, and he was fined for adulteration, for selling coloured milk and also for failing to have his name and address on the cart. The fines and costs amounted to £31 10s. The milk from the farm, when he received it, was found to be of satisfactory quality.

Although an officer cannot take official samples outside his own area, *M'Nair v. Cave* (1903; *B.F.J.*, 1902, 245), if his Authority be

a purchaser in another district, he may take samples and prosecute as a private purchaser (*B.F.J.*, 1927, 7).

BRIBERY. A serious view may be taken of any attempt at bribery. A vendor who offered an inspector money to induce him to abstain from having samples of adulterated butter reported on was sentenced to two months' hard labour (*L.G.B. Report*, 1912).

NOTIFICATION TO VENDORS THAT A SAMPLE IS GENUINE.

A question which has excited some debate is "Should a vendor of a genuine sample be informed that it is genuine?" In Birmingham, the practice is to inform vendors of formal samples of *milk* that they need not keep their part any longer. Some authorities send a certificate for all cases, others a printed slip stating the sample is pure or genuine, or that there will be no proceedings. In some districts the information is only given on application. Some Authorities have discontinued sending such notices, as it was found they were used for covering dishonest practices, as certificates of excellence.

CHAPTER III

ADMINISTRATION. (iii) RECORDS

Sampling officers' records. Public analyst's records. Analytical records and memoranda.

SAMPLING OFFICERS' RECORDS

PARTICULARS are here given of the methods which have been used in Birmingham for some years and found useful.

(i.) Sample books have been previously mentioned (p. 17). Each opening is numbered, and has three detachable gummed labels. In it are entered particulars at the time of purchase (see p. 564), and if the sample is taken from a shop, cart or station, and if from a shop where the article is served from. Any label on receptacle should be copied, and notes made of any declaration or remarks by the vendor. These books are of convenient size for the pocket ; each book covers 50 samples. They are useful for preparing reports on cases.

(ii.) Daily records book. This is the chief book, and contains, as well as the particulars from the sample books, the results of the analysis, notes of prosecutions and cautions, and references to related samples (see p. 568).

To facilitate reference to the above book, five alphabetical indexes are kept. In each case the date is given, and the total number of samples taken on that date. (iii.) Farmers. (iv.) Milk carts. (v.) Chemists and drug stores ; the name of the article bought is given. (vi.) Licensed houses, the name of the licensee, and of the owner are recorded as well as the name of the article. (vii.) Street index. The total number of samples, other than those dealt with in the previous indexes, is entered, but not details either of the articles or the street number. This index is also useful as an indication whether the sampling in a particular street has been neglected or overdone.

In addition to these, alphabetical registers are also kept of (viii.) vendors prosecuted, and (ix.) vendors cautioned. In these, the samples' numbers and the dates are entered.

It will be seen that by these indexes references can at once be made to the samples for which any vendor has been prosecuted or cautioned, and with a little further trouble his previous record can be examined.

The certificates received from the Public Analyst for samples for which no prosecutions have been instituted are filed in order of date,

and kept for a few years. Prosecution certificates are filed with the particulars of the case (see p. 565) and kept for a much longer period.

Although the responsibility for the form of a summons does not lie with a Sampling Officer, it is usually a convenience for him to prepare a draft summons, and it is a good plan for him to file for reference a copy of the first summons for each particular offence. Some Birmingham forms are given in the Appendix (p. 574). If the nature of the offence is clearly shown, numerical details are superfluous, as they are given on the accompanying certificate. Two copies should be made of the summons, one to be retained and the other to be handed to the Magistrates' Clerk.

PUBLIC ANALYST'S RECORDS

The method which has been used in Birmingham for many years is that with each sample, or number of samples, the Sampling Officer shall bring a certificate form, either "Informal purchases" or "Vendor notified" (A or B, see p. 570), upon which they are entered. When the analysis is finished the sheets are completed by the analyst and returned to the officer. If one sample of a number is adulterated, it and any related samples are struck off that sheet and entered on another sheet. For formal samples, for which a prosecution appears possible, a statutory certificate (C or D, see p. 572) is filled up.

The number of samples of each article received is entered in the "Total Samples Book" (B, see p. 568), classified under four headings, and in due course the number adulterated is filled in. Each opening of the book is reserved for a definite period—a month or quarter, according to the size of the district—and the right-hand columns are used for a summary of the period for statutory reports. Each sampler has his letter (and a corresponding seal to match), and the last number of each batch is entered so that any numbers missing would be noticed.

Any condemned samples are entered in the "Adulterated Samples Book" (C, see p. 569), with a copy of the certificate given for each, and in due course the action reported by the sampler to have been taken in relation to them—caution, prosecution, further samples, etc. At the end of each quarter, the book is reviewed to ascertain if the adulterated samples are being followed up; and if not, why? There should be a definite decision as to action, or no action, on each adulterated sample.

In each of these books it is useful to enter informal samples in red ink and formal samples in black ink.

For the statutory Quarterly Reports, Government circulars have suggested that one line shall be given for each sample analysed, but

such a course is sheer waste of time. The Ministry of Health is quite satisfied if the total number of samples of each article, distinguishing formal, informal, and private samples, is given with a statement as to the number adulterated and particulars of the nature of the adulteration in each case. (Cp. Ministry of Health Memo. 36/Foods, January, 1929.) There is no obligation on a Public Analyst to include any account of the action taken in regard to adulterated samples, but it is better that he should include such information and not leave such facts to be sent to the Ministry by the Town or County Clerk. If there appears to be slackness, the Ministry may very properly enquire why there has been no action on certain samples.

Although the Ministry is entitled to full particulars as to action, if the Quarterly Reports are printed and circulated, it may be inadvisable to inform possible adulterators by them as to cases in which no action, perhaps for very good reason, has been taken. In such cases, particularly with regard to milk, a covering letter giving further details, may be sent to the Ministry with the Report. All the necessary facts should be obtained from the "Adulterated Samples Book."

When samples of milk are taken by samplers from the cows at farms they should *not* be included in the number of milk samples for the Quarterly Report. It is obviously absurd to include in adulteration statistics a number of samples, which, whatever their composition, are technically "Genuine, as obtained from the cow." Such samples are conveniently entered in green ink in the "Miscellaneous Book" (D, see p. 569), in which are also entered samples from various Corporation departments, and any samples of food or drugs that are not bought under the Act.

ANALYTICAL RECORDS

As the decision as to the genuineness of a sample depends on comparison with other samples, it is obviously useful to tabulate the analytical results for each article on a separate sheet. Foolscap sheets ruled in squares, about three to an inch, are satisfactory for a small or moderate number of samples of an article. For larger numbers a foolscap book ruled as required, or a book with printed headings is better. The sheets are kept in files, the indexes of which include the names of particular articles, as well as letters of the alphabet, being written on blank indexes according to requirements.

When articles are received for analysis, their numbers are copied on the corresponding sheets in red, black or green ink, as mentioned above. If there are several assistants, it is convenient to have a small drawer for each, and to put the sheets belonging to articles

given out for analysis into the particular assistant's drawer, until the results are given in to the Public Analyst.

ANALYTICAL MEMORANDA

I have found that the most expeditious way of indexing analytical periodicals is to underline any useful part and then let the clerk copy the underlined part with the reference, and the author, on foolscap paper, with the length in inches of the article. These sheets are subsequently cut into strips and stored in envelopes in a foolscap file. When there is an accumulation on a particular subject, the strips can be stuck together, and subdivided if necessary. The indexes of these files also should contain names as well as letters. Analytical methods, cuttings, and memoranda are also kept in the files.

It is necessary to make rules for indexing as one goes along, to make notes of them, and to stick to them, otherwise, for example, there may be entries both under "Calcium" and "Lime." Some general headings, as "Alkaloids" and "Preservatives," may be useful.

When in a journal there is a reference to a previous article, it is useful to put a reference on the earlier article to the later one.

CHAPTER IV

PUBLIC ANALYSTS' CERTIFICATES

Requirements. Personal responsibility. Decomposition. Facts, not opinions only. Examples. Definite figures. Fair statements. Solids with liquids. "Added" water. Milk certificates. Authority of the British Pharmacopœia. Seven classes of drugs. Limit of error. Weight of sample. Observations.

THE almost unique position held by the certificate given under the Food and Drugs (Adulteration) Act is not always realised. In an ordinary criminal prosecution, witnesses give evidence as to the alleged offence, and any doubtful points can be cleared up by questions. This Act, however, has decided that the written certificate may take the place of verbal evidence, and therefore, if it is incomplete, ambiguous, or incorrect in form, the prosecution will probably be dismissed, although the analysis of the article is quite correct, and an offence has really been committed.

Ultimately the Town Clerk, or other prosecuting authority, is responsible for the *form* of the certificate, and the Public Analyst for the *figures* on it. A compromise is often necessary, as the one may desire impossible figures to be given, and the other may prefer a form which is legally unsatisfactory. A public analyst should know enough of the law to see both sides and give a certificate both legally and analytically correct.

REQUIREMENTS. Consideration of the form given in the schedule of the Act indicates that the certificate must give evidence on three or four points: (1) The identity of the article with that purchased from a particular vendor, thus supplementing the evidence of the Sampling Officer. (2) That it has been analysed by the properly appointed public analyst. (3) In some cases, that any deficiency in it has not arisen between purchase and analysis. (4) The composition of the article, and possibly the reason for any addition.

PERSONAL RESPONSIBILITY. From these considerations practical points arise. A chain of evidence should be complete, and it is desirable that the Sampling Officer should personally submit any formal sample to the Public Analyst, or send it by registered parcel post, in which case the Public Analyst should personally open it. There are often special circumstances in the taking of a particular sample, which by personal contact can be discussed. If an assistant officer or analyst transmits a sample, his name should be put on the certificate, and he should by ready

in a prosecution to give evidence of his non-interference with the sample while in his charge.

The requirements of sect. 15 as to the "competent knowledge, skill and experience" of a public analyst, and the necessity of his appointment being approved by the Minister of Health (cp. Ministry of Health Memo. 36/Foods, January, 1929), indicate that the certificate shall be the work of a properly qualified and approved person, and that, therefore, the words on the certificate "I received" and "my analysis" are personal and not by deputy. If any public analyst considers his assistant's analysis is sufficient, let him truthfully fill up his certificate and instead of "my analysis" write "the analysis of my assistant," and observe if a conviction follows. The writer believes, and has always practised, that the analytical figures relating to a sample shall be the actual determinations of the one who signs the certificate. Further, he should be in a position to prove every stage; for instance, if a sample of vinegar is deficient in acetic acid, not only should the Public Analyst have determined its strength, but also checked the accuracy of any pipette used for measuring the vinegar analysed, as well as the strength of the standard alkali used in the determination. In these conditions only does the certificate bear the Public Analyst's personal guarantee of accuracy, and he can face with confidence any cross-examination on the subject.

The question was raised as to the sufficiency of the Birmingham Public Analyst's participation in the analysis in the Appeal Case *Bakewell v. Davis* (1894). The Analyst's practice was to see the milk and the flask for fat weighed by his assistant (myself), and pipette the milk on the paper coil for fat. The extraction, evaporation and drying were then attended to by me, and the Analyst finally saw the solids and fat weighed. This was probably the bare minimum necessary, but the Analyst had personal knowledge of the essentials of the analysis and the Appeal Court upheld the conviction. In September, 1899, a case was dismissed at Southwark because the analysis was entirely done by an assistant, though the Public Analyst signed the certificate. This Act differs from the Fertilisers and Feeding Stuffs Act of 1926, where analysis by a deputy is sanctioned.

It is of course advisable that two persons should examine any adulterated sample, when there is sufficient of it, and agreement between a principal and his assistant is more valuable when the work is entirely independent, and the results will give an indication of the error of the process. Mistakes may, and do, happen, but it is impossible that two persons will make an identical mistake, and agree in an error.

DECOMPOSITION. The third point—decomposition—has produced much argument. A Middlesex Quarter Sessions case

(*Peart v. Barstow, Analyst*, 1880, 5, 212) decided that "Fresh when delivered" was not sufficient. It was prominent in a lime-water case (see p. 454) in 1898. If the statement "no change" be applied *without any time limit*, to an article liable to evaporation, it may be misunderstood to cover the whole of the time since the article was made. The writer has for years used for milk the statement "No decomposition had taken place in the article that would interfere with the analysis," and for other articles "No change had taken place in the article *since purchase*, that would interfere with the analysis." This meets the obvious intention of the Act, and the statement has been justified by the examination being commenced within as short a period as is possible after purchase. Any question as to which articles are "liable to decomposition" has been avoided by having the statement printed on *all* prosecution certificates.

The writer is not aware of any prosecution for a decomposed milk, but it appears to him that in a flagrant case of milk adulteration a careful statement as to the amount of possible decomposition would not prevent a conviction, particularly if the calculation of adulteration were based on the percentage of nitrogen present.

The references to milk and butter in the schedule suggest that there was little thought of "decomposition" except that the analyst should report if a milk was sour, or a butter rancid. The question has been discussed as to whether "decomposition" is limited to that caused by bacteria, as milk and butter, or also includes chemical decomposition, as lime-water, or simple evaporation, as spirits (see also p. 494).

An Appeal Case on the question was *Hudson v. Bridge (C. & D., 1903, March 28, April 4; Analyst, 1903, 28, 165; B.F.J., 1903, 84)*, where it was thought that a statement ought to be made for substances liable to decomposition, and that "interfere with the analysis" does not mean prevent the possibility of accurately analysing the substance, but prevent the analysis being effective for showing what was the constitution of the article at the time of sale.

FACTS, NOT OPINIONS ONLY. The right of a public analyst to fix a standard or limit of composition has often been challenged, and sometimes successfully. This is rather curious, as without that right the Act would be futile except for the few articles for which there are statutory standards, though some adulterators would rejoice at it. In an egg substitute powder case it was suggested that there could be no standard for a "substitute." It is obvious, however, that a substitute must not be worthless, and that comparison with other samples might make a standard.

An analyst, not being in possession of the full facts of the case, cannot, and must not, presume to say an offence has been committed; that is the province of the magistrates. The certificate must,

however, state the facts of the analysis sufficiently to enable the magistrates to decide for themselves that the provisions of the Act have been infringed (*Fortune v. Hanson, Analyst*, 1896, **21**, 53). The certificate should contain evidence and not only the conclusion at which the analyst has arrived. Also "I estimate" was accepted as correct, *Newby v. Sims* (1894). See also *Lee v. Bent, B.F.J.*, 1901, 196; *Jenkins v. Naden* (1919); *Bowker v. Premier Drug Co.* (1927); and *Bayley v. Cook (Analyst)*, 1905, **30**, 107). The latter calls attention to another aspect of the subject. There should be fullness and simplicity in certificates so that the defendants should know what charges they have to meet, and that they may discover the standard of comparison without any elaborate calculations.

When, in the witness-box, the writer has been asked the authority for a standard, he has stated that he has fulfilled his duty in making one, endeavoured to prove to the magistrates that it is reasonable, and asked them to accept it.

The relation of the magistrates to the analyst's standard is discussed below (p. 52).

The problem for the analyst is to give a certificate that shall be his deputy and give the necessary facts of evidence in as simple a form as is possible.

EXAMPLES (cp. Appendix, pp. 572 f.). Before the chief part of the certificate, the following words are printed in the schedule :—

"I am of opinion that the said sample contained the parts as under, or the percentages of foreign ingredients as under."

Leaving the whole of this wording in the certificate does not invalidate it, *Bakewell v. Davis* (1894), but it is better to cancel parts of it according to the statement of composition, as is shown in the following examples, all of which have been used in successful Birmingham prosecutions. Sect. 17 (3), "or a form to the like effect," gives authority to vary the certificate.

I. I am of opinion that the said sample was—(1) Artificial vinegar. (2) Margarine, the fat of which did not contain more than 2 % of butter fat.

II. I am of opinion that the said sample contained the parts of foreign ingredients as under :—(1) Boron preservative equivalent to 33 grains of boric acid per pound (Sponge Cakes). (2) Formic aldehyde 2 parts per 100,000 (Milk).

III. . . . contained the percentage of foreign ingredients as under :—(1) Chicory 65 % (Coffee). (2) Oil other than almond oil of the B.P. 100 %. (3) Vegetable matter foreign to chicory 100 %.

IV. . . . contained the percentages as under :—(1) Fat 3·5, Solids-not-fat 7·6, Water 88·9, Total 100 (Milk). (2) Ash soluble in water 1·8, Ash insoluble in water 1·2, Moisture 11·2, Organic constituents 85·8, Total 100. Cold water extract 6·6 % (Ground ginger). (3) Acetic acid 3·4, Solid colouring matter 0·4, Water

96.2, Total 100. The sample was weak artificial vinegar. (4) Whisky of 35 degrees under proof 92, Excess water 8, Total 100.

V. . . . contained parts as under :—(1) A mixture of about equal parts of butter and margarine. (2) Quinine sulphate (B.P.) 127 grains and diluted sulphuric acid (B.P.) 210 minims per 6 oz. bottle (Quinine mixture). (3) Lime-water (cp. p. 455).

Lime (CaO)	0.000	gramme	per	100	millilitres
Calcium Carbonate (CaCO ₃)	0.006	„	„	„	„
Water.	sufficient	to	make	„	„

In some cases, as tinctures, it may be convenient to express all the constituents as gm. per 100 ml., when the total (*i.e.*, w/v) will be not 100, but 100 times the sp. gr.

To state the percentage of foreign ingredient is sufficient when a substance which could not or ought not to be found is present (as, sand in sugar), but when the foreign ingredient is something which is ordinarily found as a constituent of the article, the difference should be shown in the certificate, *Fortune v. Hanson* (1896). “Water 18 %, fat, curd, etc., 82 %,” is an example of the latter, and there is no need to give separate figures for curd and salt, unless they are in excess. According to *Jenkins v. Naden* (1919), the certificate should set out all the matter complained of, but all the figures of the analysis need not be given. A prosecution for liniment of turpentine deficient “50 % in camphor and turpentine” was dismissed because the deficiency of *each* was not stated (*Analyst*, 1930, **55**, 752; *P.J.*, 1930, Oct. 25).

In *Rudd v. Skelton Co-operative Society* (B.F.J., 1911, 68), the certificate for lardine stated “Water 25 %, Fat, etc., 75 %. Observation. Adulterated with 25 % of water.” Objection was made to the use of the word “adulterated,” but the High Court accepted it, being unable to distinguish between foreign ingredients and adulteration.

DEFINITE FIGURES. One essential requirement of a certificate is that it shall contain definite figures; “contains arsenic” and “contains a serious quantity of arsenic” have each been ruled as insufficient, *Lee v. Bent*, *Barlow v. Noblett* (both 1901, B.F.J., 1901, 196). A prosecution for calcined magnesia at Glasgow was dismissed which quoted the B.P. standard as “only the slightest reaction for lime” as being too indefinite to fix an exact standard. Had a commercial standard been given, a conviction might have followed.

While “5 % of added water” was ruled to be insufficient (*Fortune v. Hanson*, 1896), the following were considered satisfactory: Brandy, “reduced from 25° under proof to 27.6° under proof,” *Findley v. Haas* (B.F.J., 1903, 87). “Margarine containing only 3 % of butter,” *Allwood v. Gregory* (B.F.J., 1905, 177). “Normal

vinegar contains at least 4 % of acetic acid," *Robinson v. Newman* (B.F.J., 1917, 154).

When adulterations are given as percentages, the statement should be unambiguous; there should be no doubt as to whether the figure is a percentage of a standard, or of the article itself. In a recent prosecution a certificate is reported to have stated "Ethyl hydroxide 14.44 per cent. by volume, corresponding to a deficiency of 9.7 per cent. of that ingredient." According to this the proper percentage might be either $(14.44 \div 9.7 =) 24.14$, or 16.0. A deficiency of fat in milk is usually expressed as a percentage of the *minimum limit* (3 %), while excess of water in butter is usually the percentage of water over 16 %, in the *butter itself*.

It is sometimes more convenient to use "parts" instead of "percentages." The statement that about one third of a sample of coffee is chicory, avoids too precise a statement of a somewhat indefinite figure.

FAIR STATEMENTS. Certificates should be a fair statement of the facts, and no attempt should be made to magnify the offence by the form of statement. Some certificates which have been given for copper in peas appear to err in this direction. The copper is largely, or entirely, in combination with the organic matter of the peas, and therefore, to state that copper sulphate is present, or to state "copper, equivalent to — copper sulphate," are both undesirable. The first is chemically incorrect, and the second is misleading. Further, multiplication of the copper by 3.9 is an unjustifiable magnification of the adulteration. The real offender is the copper, and not anything in combination with it.

SOLIDS WITH LIQUIDS, ETC. A tin of preserved peas contains both peas and liquid, and a fair proportion of each should be given to the analyst. The liquid must not be ignored, as the analyst must certify the composition of the sample and not the solid part of it only. This question has been discussed at a meeting of the Society of Public Analysts (S.P.A., 1897, 22, 145). To express the copper on the whole sample would be misleading as some housewives throw the liquid away; that also assumes that the sampler's division is exact. Peas and liquid have a different content of copper. To meet these difficulties, the following certificate is suggested:—

"The said sample contains the parts as under:—

Strained peas	.	.	.	— oz.
Liquid	.	.	.	— oz.
				—
Total	.	.	.	— oz.

— grains of copper, in combination, were present per pound of strained peas.

Observations. The copper was added to improve the appearance

of the peas. The addition of compounds of copper for colouring food is prohibited by the Public Health (Preservatives, etc., in Food) Regulations, 1925." Similar considerations apply to "bread and butter" and other heterogeneous samples.

"ADDED" WATER. It is undesirable to use on certificates statements which cannot be proved, such as "added water"; an officer may say that if he has seen it actually done, but not an analyst; better expressions are "excess" or "extraneous." To *assume* that a deficiency is due to the addition of water, and certify it as a *fact*, becomes an absurdity if the defence can prove the deficiency is due to *evaporation* and not to dilution. This occurred in a whisky case (*Smith v. Jeffreys*, 1897), where the analyst certified the presence of "added water," and the defendant was charged with "selling whisky under strength after diluting it with water." It was proved that there had been no addition of water but that the spirit had been in stock fifteen months. The offence charged was obviously not proved and the prosecution was dismissed (*F. & S.*, 1897, March 13, May 29).

A prosecution for milk containing "11 % of added water" was dismissed, apparently, partly because of the word "added" and partly because the fat present was above the average (*B.F.J.*, 1930, 59).

Certificates for spirits may be found in several appeal cases, viz. :—*Newby v. Sims* (1894), *Findley v. Haas* (1903), *Ross v. Helm* (1913), and *Preston v. Grant* (1925). The following form gives definite information to the magistrates and also to the publican, who is familiar with degrees under proof :—"Whisky of 35 degrees under proof 92, Excess water 8—total 100. The above opinion is based on the fact that the sample was 40·4 degrees under proof."

MILK CERTIFICATES. These require special consideration. While it is not essential to give the percentages of fat, solids-not-fat and water (*Bridge v. Howard*, 1897; *Analyst*, 1896, **21**, 305; *Bayley v. Cook, Analyst*, 1905, **30**, 107; and *Jenkin v. Naden*, 1919), it is better to do so, so that the magistrates may compare the percentages of fat and of solids-not-fat with the Regulations limits. My certificates which have been unchallenged for many years stated, "Comparison of the above results with the minimum limits fixed by the Sale of Milk Regulations, 1901, viz., Solids-not-fat 8·5 %, Fat 3 %, shows that the said sample of Milk was deficient of — % of the said minimum amount of solids-not-fat (or fat)." Such certificates are a scientific statement of fact, which can be proved, and not a theoretical, sometimes unprovable, opinion like "added water." They are equally true if the deficiency is due to adulteration or to natural causes. If both constituents were below the limit, and the fat deficiency was less than that of solids-not-fat, the former

was not mentioned so that a defendant should not think he was accused of skimming as well as watering, when the addition of water would account for the deficiency of fat. If the deficiency of fat was the greater, for example, deficiencies of 10 % of solid-not-fat, and of 20 % of fat, the statement " of which 10 % was due to the addition of water " was added to the fat deficiency. See also *Dearden v. Whiteley* (1916) (cp. p. 572).

It is not always realised that a high fat in a milk acts in the same direction as addition of water, and that the adulteration may be overstated. Such an excess of fat might be due to a retailer selling the top-milk, hoping excess of cream would cover added water, or to a farmer putting extra strippings to his watered milk. In 1918 a sample of milk had solids-not-fat 7·8 %, fat 5·8 %, water 86·4 %. After stating that the deficiency of solids-not-fat was 8 %, as before, the observation continued, " If the amount of fat in this milk be reduced to 3 %, the amount of solids-not-fat would be raised to 8·02 %, and the deficiency of solids-not-fat would then be $5\frac{1}{2}$ %." The vendor was fined £5. The only fraction used in milk deficiency has been one-half, and that only in small adulterations.

The decision *Hunt v. Richardson* (*Analyst*, 1916, **31**, 224 ; *B.F.J.*, 1916, 327, 343, 357), in which 28 cows, previously milked at 1 p.m., gave at 5 a.m. milk containing 2·73 % of fat, and 8·97 % of solids-not-fat, has placed milk in a unique position. The critical question is now, not " Does a sample come below the presumptive limits ? " but " Is it as it came from the cow ? "

Consequently a sample below the presumptive limits may be genuine, but also, which is often overlooked, a sample above these limits may be adulterated, when compared with samples of milk taken by officers at the farm. If this were strictly followed, only a few samples of very good milk could be passed as genuine unless a comparison sample were taken at the farm. This is of course impracticable, but when the farm is being visited I have preferred keeping back my certificate until the farm milk has been analysed. Should the farm milk resemble the suspected sample, the latter is passed as genuine, but if not, as often happens, a further comparison may be added to the certificate. A station sample had solids-not-fat 8·1 %, fat 3·0 %, indicating a deficiency of $4\frac{1}{2}$ % of solids-not-fat. The following addition was made : " Comparison of the above results with milk taken at the farm, containing 8·8 % of solids-not-fat and 4·0 % of fat, shows that 8 % of added water was present in the said sample, and that 17 % of the original fat was deficient."

In 1924 a sample of evening milk taken at a Birmingham railway station contained 3 % of fat and 8·6 % of solids-not-fat, both being above the limit. The farmer was prosecuted, the certificate stating, " Comparison of the above results with milk containing 8·9 % of solids-not-fat and 4·4 % of fat shows that the above sample

was deficient of 3 % of the said amount of solids-not-fat, and 32 % of the said amount of fat." No objection was taken to the certificate, but owing to the sample being taken at the week end, there was an interval of five days between it and the farm sample. The case was withdrawn and the farmer fined £5 for a sample of morning milk containing 2.6 % of fat taken at the same time as the evening sample.

THE AUTHORITY OF THE B.P. This is an important question in relation to certificates for adulterated drugs, and it is convenient to consider it here, as well as to give an outline classification of drugs from the analytical standpoint. There have been discussions, preceded by papers, on the matter at the Society of Public Analysts (*S.P.A.*, 1901, 26, 86) and at the British Pharmaceutical Conference (*B.P. Conf.*, 1913, 576-617).

By an Order in Council in 1851, it was made an offence to make any compound or distilled medicine contained in the London Pharmacopœia except according to its directions, unless by special medical prescription. This was limited to England and Wales. The Medical Act of 1862 substituted the British Pharmacopœia for that of London, and it is still in force. The Pharmacy Act of 1868, sect. 15, gave similar provisions in relation to the B.P., and fixed the penalty as £5. It should be noted that the offences are "*compounding*," and as there are no references to *selling*, they are consequently of little practical use.

There have been three important appeals. *White v. Bywater* (1887) decided that a person asking for tincture of opium is entitled to receive the B.P. preparation (see p. 501). *Dickens v. Randerson* (1901) decided that the B.P. was *primâ facie* evidence as to the composition of articles contained in it, and asked for under the B.P. name (see p. 529). In *Boots Ltd. v. Cowling* (1903) it was further ruled that magistrates must consider other evidence besides that of the B.P., if it be offered (see p. 535).

From this it follows that the B.P. is not an *absolute* but an important *presumptive* standard. Errors have appeared in the B.P., and if it were an *absolute* standard vendors might be fined, not for their own wrongdoing, but for a mistake in the B.P. Further, a work of that kind can never be up to date, and to pass a drug as genuine, which contained an adulterant unknown at the time of the publication of the B.P., would be absurd, though there was much argument over arsenical sodium phosphate in 1900 (see p. 476).

In addition to the current B.P. reference may sometimes be necessary to the older ones, as preparations of the 1885 and 1898 editions are still in use. Drugs and preparations outside the B.P. may often be found in the British Pharmaceutical Codex, which is another presumptive standard, and sometimes reference to Squire's "Companion to the B.P." or Martindale's "Extra Pharmacopœia"

may be useful, as well as papers in the pharmaceutical Press, the *Analyst*, and in volumes of the *Year Book of Pharmacy*.

A practical question is, "Should the B.P. or other authority be quoted in a certificate?" The writer considers this to be undesirable as a general rule, and has often preferred to say the article "should contain," without giving any authority for his standard. If challenged he has been prepared to give other reasons for his standard, particularly the analyses of other samples, and so avoids it being judged by the B.P. only (cp. p. 483).

On the other hand, it may be convenient to quote the B.P. in cases where it may be difficult to definitely name the adulterant. A certificate "100 % of oil other than Almond Oil of the B.P." resulted in a fine, while "Peach Kernel Oil 100 %" was dismissed, the actual adulterant being apricot kernel oil.

At other times reference to the B.P. may make the certificate clearer to the magistrates, as in this example:—"Borax (B.P.) 15, Glycerin (B.P.) 63, Water 22, Total 100. Glycerin of Borax prepared according to the B.P. contains:—Borax (B.P.) 11·7, Glycerin (B.P.) 88·3, Total 100."

SEVEN CLASSES OF DRUGS. From an analytical point of view, drugs may be divided into the following classes:—

(1) *Simple drugs, commonly of vegetable origin.* Usually the B.P. is a satisfactory standard for this class, though the wide variation in the price of some drugs might cause difficulty. One quality of ground rhubarb, for instance, is three times the cost of another. The use of the word "best" in the purchase might enable action to be taken under sect. 2 (1), "not of the quality."

Some drugs are also foods, and there may be a commercial quality which is neither. Gum acacia may be a drug, or used as a food in confectionery, or sold for commercial purposes, and in that case is outside the Act (see p. 47).

Saffron is one of the drugs omitted from the 1914 B.P. Its medicinal value is doubtful, but it is expensive, and dyed calendula florets should not be substituted.

It has been argued that the B.P. descriptions of natural products only is binding, and not the "Characters and tests" (see Cod Liver Oil, p. 274).

(2) *Salts and other chemical preparations.* The most frequent fault in this class is the presence of impurities, particularly arsenic and lead, and great care should be taken before a drug somewhat below the B.P. limit is condemned as adulterated. Self (*P.J.*, 1922, Nov. 18) has compiled an interesting table which shows how much arsenic and lead would be taken in a *year* if the maximum dose of the drug containing the 1914 B.P. limit amount of these impurities were taken three times a *day*. Taking two common drugs, tartaric acid and bicarbonate of soda, the annual arsenic taken under the above

conditions would be $\frac{1}{32}$ and $\frac{1}{15}$ grain, respectively, or half, and one *single* maximum dose of arsenic, respectively. The annual lead doses would be $\frac{1}{2}$ and $\frac{1}{6}$ grain, respectively, or one minimum *single* dose, and $\frac{1}{3}$ dose, respectively. The following chemicals have commercial uses as well as medical ones: ammonium carbonate, calcium chloride, chlorinated lime, copper sulphate, ferrous sulphate, potassium carbonate and bichromate, and must not be *assumed* to be drugs. Medicinal alum, borax, and nitre are now each officially described as "purified." "Saltpetre" is not now mentioned. "Liquefied carbolic acid" is the B.P. name. "Carbolic acid" is a disinfectant, and I know of no decision that a disinfectant is a "drug."

(3) *Synthetic and organic preparations.* For some of this class the B.P. can hardly be quoted, as usually the common name is not given in the B.P. "Aspirin" is *not* given as a synonym for acetylsalicylic acid. Quinine sulphate is one of the few alkaloidal preparations that can be bought without difficulty; for other alkaloidal preparations a prescription is usually required, and even then it may be difficult to obtain enough without arousing suspicion that it is for analysis.

(4) *Compound drugs for which the B.P. gives tests.* Sweet nitre, tincture of opium, and syrup of ferrous iodide may be taken as examples. In prosecutions for articles in this and the next class, the defence has sometimes been that the B.P. article is little used, but that the substituted article sold is in popular demand. Such claims may be correct if the B.P. formula is unsatisfactory, and the analyst should ascertain of there be any competing formula.

One prosecution for lime-water was dismissed because the certificate stated "26 % of lime had been extracted."

(5) *Compound drugs for which the B.P. only gives directions for making.* This is a large and important class for which the B.P. is of limited use for deciding the correctness of the product, except by making it. Even with simple preparations it is not safe to assume that the composition of the product can be calculated from composition of the constituents. There are three difficulties, filtration, evaporation and decomposition.

A definite amount of quinine hydrochloride is used to make quinine wine, then follows "filter if necessary." For a weak wine the defence may be, rightly or wrongly, that part of the alkaloid had been filtered out, and the analyst must be prepared for that defence.

Ammoniated tincture of quinine has a definite amount of ammonia used in its preparation, but there will be some evaporation, and a standard must be established by the analysis of samples, which is lower than the calculated composition.

The disappearance of acetic acid from vinegar of squill gave rise to the appeal case *Hudson v. Bridge* (1903), but the 1914 B.P. gives a limit for acidity.

The important thing about a drug is, of course, that it shall be of its full therapeutic value ; variations in the solvent are therefore of less importance than in the active constituents. In the case just mentioned it was argued that the acetic acid had served its purpose as a solvent, and therefore its presence or absence did not matter. A constituent of the solvent may be necessary, not only to dissolve, but to retain active matter in solution, also the solvent may be of medicinal value. For the substitution of glycerin for alcohol see liquid extract of cascara sagrada (p. 543). In one remarkable case evidence was given that a deficiency of 42 % of alcohol in tincture of myrrh was not to the prejudice of the purchaser ! Reduction in the amount of alcohol in a preparation may facilitate illegitimate competition in price.

The B.P. directions, which may be quite good for the preparation of small quantities, may be impracticable on a manufacturing scale, but may produce a better article. Manufacturers often claim special value for their preparations. When such preparations differ seriously from the official one, they must not be sold as B.P., but with an appropriate label. An oxymel of squill prepared from glucose syrup, without declaration, is an example.

The following certificate may be taken as an example for this class : " Camphor 5, Colza Oil 95, Total 100. Camphorated oil should contain :— Camphor 20, Olive oil 80, Total 100." The vendor was fined £5.

One analyst gave a certificate for Gregory's powder on which he said it was not in accordance with the B.P. As that work is not a legal standard, and he did not say it was not genuine, the summons was dismissed (*B.F.J.*, 1900, 27). Another case was dismissed because the analyst omitted to mention fennel fruit as a constituent of compound liquorice powder, while mentioning all the other ingredients (*P.J.*, 1902, June 14).

(6) *Compoundeds drugs prepared according to a prescription.* These are dealt with in detail in a later chapter (p. 546). The following certificate is an example in which there is an excess of ammonium carbonate as an ingredient, which is not of definite composition, and for which no upper limit is prescribed. The vendor was fined : " Ammonium carbonate at least 70 grains in 8 oz. of weak infusion of senega. The mixture contained 22·8 grains of ammonia, and 14 grains of solid extract. The prescription ordered 40 grains of ammonium carbonate in 8 oz. of infusion of senega. 40 grains of ammonium carbonate should not contain more than 13 grains of ammonia. There was, therefore, an excess of at least 75 %. 8 oz. of infusion of senega should not contain less than

42 grains of solid extract. There was, therefore, a deficiency of not less than 57 %."

(7) *Proprietary medicines, and similar preparations.* Proprietary medicines, as "Murray's Fluid Magnesia" (p. 546), and vaseline, are specifically exempted from sect. 2, and in the absence of any standard could hardly be certified under sect. 1 (1) (b). There are also medicinal preparations in popular use, such as Composition powder; Glycerine, honey and lemon; and Tincture of myrrh and borax, for which no proprietary rights are claimed, but which each manufacturer makes according to his own formula. For these things it would be difficult to establish a standard and condemn any as adulterated. Action for "false label" is, however, possible, if the label indicates drugs which are absent. Some articles which claim to contain vitamins may also be included in this class. A conviction has been obtained for cod liver oil tablets (see p. 275).

LIMIT OF ERROR. It is not unusual to consider drugs to be genuine which do not vary more than 10 % above or below the standard figure. This must not, however, be taken as always applicable. A variation of 10 % in the vehicle is often less important than in an active principle, also, while a deficiency of 20 % might suggest fraud, an excess of 20 % will not usually do so, though sometimes the active ingredient is the cheapest. If the absolute amount be not considered, a percentage figure may be very misleading. If the alkaloid, or active principle actually weighed, only amounts to a few mgm., the analytical error itself might be 25 % (cp. p. 550). On the other hand, an error of 10 % in dispensing 480 grains of magnesium sulphate would be inexcusable.

In the analysis of compounded drugs, including prescriptions, the limits given by the B.P. for the constituents must be considered, and it must not be assumed they are of 100 % purity. When such a range is given, an allowance of 10 % below the minimum, or of 10 % above the maximum, may be quite unnecessary. The comparison of the figures obtained with several samples of a drug purchased at the same time is often very useful.

WEIGHT OF SAMPLE. The form of certificate in the Schedule provides for the weight of the sample to be inserted unless it cannot be conveniently weighed, and before 1901 cases were dismissed because the weight was not given. In that year *Sneath v. Taylor* (*Analyst*, 26, 1901, 167) decided that the omission did not render the certificate invalid, unless the accuracy of the analysis depended in any way on the weight of the sample. For seidlitz powders the weight should be given, and in some other cases the weight may be useful as giving some indication of the quantity the officer received for his money.

OBSERVATIONS are only to be made when the case is one of adulteration, and such observations as "the abstraction of fat is a

fraud, and may be injurious to health," though unauthorised as suggesting abstraction, which was disproved, did not render the certificate invalid, *Bakewell v. Davis* (1894). An analyst has no right to report extraneous facts unconnected with the analysis, and if he does, his certificate would be inadmissible as evidence of such facts, *Queen v. Smith and Kerr* (*F. & S.*, 1896, April 4).

Such remarks as "colouring matter is used to impart a fictitious appearance of richness to the milk," are in order, and while a statement may be made as to injuriousness, a certificate need not make them, *Hull v. Horsnell* (*Analyst*, 1904, **29**, 387; *B.F.J.*, 1904, 233).

In a brandy case the analysis clearly showed that there was adulteration, and although remarks made on it were inconsistent with these figures, it was ruled that the error did not vitiate the certificate (*Lowery v. Hallard*, 1906).

It may be remarked that it is superfluous to print on the certificate the notes to the schedule of the Act.

CHAPTER V

PROSECUTIONS. EVIDENCE

Food or drug? Adulteration Act (1928). Sections 1, 2, 3, 4, 5, 17, 18, 30. Analysts' certificates, sections 28, 31, Schedule 2. Statement of appeal cases. Other Acts of Parliament. EVIDENCE. Fairness. "Hearsay" and other evidence. Rebutting evidence. Analyst's evidence, milk adulteration. Official sampler's evidence, samples taken at farms. Farmer's evidence.

FOOD OR DRUG? Before taking out a summons under the Adulteration Act, a preliminary question should be asked: Is the article a food, a drug, both or neither?

In a prosecution for chewing gum containing paraffin wax, labelled "This must not be eaten, but chewed only," the magistrates found that it was not an article of food to be eaten in the ordinary sense. That it might be included under "flavouring matters and condiments" was apparently not argued. The case for appeal (*Bennett v. Tyler*, 1900) was insufficiently stated, and while quashing the conviction, Mr. Justice Channell said it by no means followed that there might not be facts stated in regard to this particular article which would make the sale to be that of an article injurious to health, or to the prejudice of the purchaser. Is a "bread improver" a "food" which "ordinarily enters into human food"? (p. 126).

Usually there is no difficulty about a "food," but even with ordinary foods the question might arise. It might be pleaded that *at the price the article was sold*, somewhat sour milk was sold for pigs, and not for use "by man," that stale bread was intended for rabbits, or defective flour for making into a paste for wall paper. An appeal case decided that coffee was a "food," rather than a drink (*Analyst*, 1892, 17, 157), and a Prize Court declared it to be a "food" (*Grocer*, 1916, Aug. 5). "Soda" was held not to be a "food" (p. 462).

There are some drugs which are sold for commercial purposes as well as medicinal ones, and unless some indication is given that an article is to be used as a drug, the inferior quality may be legitimately supplied. Benzoin is used for polishes, olive oil is used as a lubricant, and linseed oil in paints. Turpentine should be bought as "rectified oil of turpentine," its B.P. name. A "best commercial quality" over 99 % pure may be dangerous as food owing to the presence of arsenic or lead. A careful vendor will attach a suitable label.

A grocer who sold beeswax containing 50 % of paraffin told the purchaser he could not guarantee it to be pure, but he sold it as

"beeswax." The magistrates dismissed the case, finding (1) that prepared beeswax is mentioned in the B.P. as "yellow wax"; (2) that in the preparations of medicines wax is used as a vehicle only, and not as possessing any medicinal properties, and that owing to the statement made at the time of sale, it was not to the prejudice of the purchaser. (3) The sale was by a grocer. Therefore, it was not the sale of a drug within the meaning of the Act. The appeal to the High Court (*Fowle v. Fowle*, 1896) was dismissed. Mr. Justice Graham thought that not one grocer in a thousand ever used beeswax as a drug; he sold it for polishing tables. He was not prepared to admit that beeswax was a drug when sold by a grocer in a country shop, and not sold as a drug. Mr. Justice Wright was of opinion that it must be in many cases a question of fact whether a particular thing was a food or a drug or something else, which the magistrates must decide in each case. Turpentine, sold to be used by a carpenter, or resin, sold to be used by a musician, ought not to be held to be drugs for the purpose of the Act, though if sold by a chemist to be used in medicine they would be drugs (*Analyst*, 1897, 22, 25).

An article sold as "arsenical soap" contained no arsenic, and for that reason it was decided not to be a drug, *Houghton v. Taplin* (*Analyst*, 1897, 22, 167). Possibly a prosecution under the Merchandise Marks Act might have been successful in preventing a fraudulent misdescription.

Although "drug," unlike "food," is not limited to that used "by man," it has been argued that ground gentian (see p. 468) and ground liquorice (see p. 469) are not drugs, as they are only used for veterinary purposes, in spite of the fact that each is contained in the B.P. Although the definition of "drug" has not been extended like that of "food," "used in the composition or preparation," distilled water has been found to be a drug (see p. 347). There appear to have been no decisions as to whether disinfectants (as bleaching powder) or dentifrices (as camphorated chalk) are legal "drugs."

There are a number of articles which are both foods and drugs—cod liver oil, olive oil, malt extract, ginger and cinnamon may be mentioned; and it may not be a matter of indifference into which class an article is put. Ginger, as a food, must be free from sulphur dioxide, but the Preservative Regulations do not apply to drugs, and a defence that ground ginger containing that preservative was sold to make "ginger tea," and therefore was a "drug" and not a "food," might be successful. Alternative summonses, one for "food" and the other for "drug," may be taken out in difficult cases, or the phrase "food or drug" may be used (p. 406).

In doubtful cases consideration must be given to the nature of the trade of the vendor, the purposes for which the article is used, the amount purchased, and the wording of any label on the package.

Although the public expects articles of a better quality from a chemist than from a grocer, it does not appear reasonable to the writer that if a mixed article be bought from a chemist, and also from a grocer, without any declaration, the chemist shall be held to be guilty of selling an adulterated article, and the grocer innocent of offence.

The olive oil of the 1914 B.P. might be too acid to be used as a food; the 1932 B.P. is to relegate this quality for use in external preparations only and is to give a standard for a high-grade quality.

ADULTERATION ACT (1928). The various offences will now be reviewed from an analytical standpoint.

Section 1. *Hull v. Horsnell (Analyst, 1904, 29, 387)* decided, what one would have thought was obvious, that the offence is not the use of an injurious ingredient itself, but its addition in such a quantity as to make the food injurious to health. A related decision (*Haigh v. Acrated Bread Co., Analyst, 1916, 41, 156*) was that if a declared ingredient (boric acid) was present in sufficient quantity to make the food injurious to health, it was "mixing" in the meaning of the section.

Wilfulness is necessary for a conviction for a sale. A defendant who satisfies the court that "he did not know, and could not with reasonable diligence have ascertained," that the food or drug was adulterated is entitled to a discharge. A vendor who had periodical analyses of the article made, would probably be credited with "reasonable diligence." In one coppered peas case, a defendant who had been told there was copper in the peas, and another, where the label mentioned copper, were held not to have exercised it (*F. & S.*, 1899, Oct. 28). A label defence (sect. 4) is not available under this section.

In the arsenic in beer case (*Goulden v. Rook, B.F.J.*, 1901, 196) it was argued that accidental contaminations were not to the prejudice of the purchaser, but that section was directed against fraudulent trade adulteration. It was decided, however, that an article injurious to health was also sold to the prejudice of the purchaser. In such cases there is a choice between prosecuting between sect. 1 and sect. 2. Medical evidence is probably advisable in prosecutions under sect. 1, except with regard to Preservative Regulations, non-compliance with which is presumed to make the article injurious to health.

Section 2. This section differs from the preceding one in that guilty knowledge is not necessary, *Betts v. Armstead* (1888).

The words "to the prejudice of the purchaser" were added to prevent prosecutions for the sale of a superior article to that asked for. Prejudice is not confined to pecuniary prejudice, or to injury from unwholesome food, but is that which an ordinary purchaser suffers when he obtains an inferior article to that paid for, *Hoyle*

v. *Hitchman* (1879). Also, the prejudice is to the purchaser in the abstract, not necessarily to a particular one, *Pearks, Gunston and Tee, Ltd. v. Ward* (1902). Extra strength in compounded drugs may increase the cost, but is still "prejudice." The sale of a pure but different article is an offence, *Knight v. Bowers* (1885), where there was a dangerous substitution, saffron being sold for saffron.

The appeal cases on the question of "prejudice" were decided, not necessarily on the facts of the case, but by the evidence submitted by the magistrates to the High Court; in one case there was no expert evidence for the prosecution, and the statement was consequently one-sided. The chief questions considered by the Judges were, "Was the article inferior?" or "Was the substitution usual?" or "Was there a standard?" Important cases are—*Smith v. Wisden* (marmalade with 13 % of starch glucose not inferior, *B.F.J.*, 1901, 382), *Hudson v. Bridge* (vinegar of squill not medicinally inferior, 1903), *Sandys v. Rhodes* (substitution of tapioca for sago, 1903), and *Anderson v. Britcher* (substitution of Mauritius sugar for Demerara sugar, *B.F.J.*, 1913, 210). References to notices of dilution of spirits are given later (p. 367). The question of prejudice in relation to milk and drugs has been previously discussed (pp. 40, 44).

There appear to be no decisions on the discrimination between "nature, substance and quality" except *Anness v. Grivell* (1915), where "quality" was ruled to be commercial quality, not description. I suggest the following application of the terms. An article is not of the *nature* when there is substitution, such as margarine for butter, or zinc sulphate for Epsom salt. It is not of the *substance* when it is mixed with foreign ingredients, such as coffee adulterated with chicory. It is not of the *quality* when there is a deficiency or a surplus of a natural ingredient, such as skimmed milk sold as milk, or butter containing excess of water, or a tincture below standard. See also "Sweet juicy oranges" (p. 310).

There appear to have been very few prosecutions for "quality." At Pontypridd a vendor was fined £5 for selling a second quality when "Best self-raising flour" was asked for. There have been fines for "New laid eggs" (p. 300). In a previous chapter it has been suggested that the use of the word "best" might at times distinguish drugs from commercial articles of the same name.

In addition to prosecutions for articles injurious to health, those for compounded drugs may also be taken under this section—*Beardsley v. Walton* (1900); *Dickens v. Randerson* (1901).

The addition of turmeric to a mixture of mustard and starch "to conceal the inferior quality thereof," or of dye to skimmed milk, would appear to be offences under this section.

It should be noted that while sub-sect. 4 incorporates the Regulations relating to condensed milk, dried milk and preservatives

relating to composition and prohibition of ingredients, no mention is made of the labelling requirement of these orders. It is therefore an offence under this section to sell full cream condensed milk containing less than 9 % of milk fat, and an offence under the Public Health Act, 1907, to sell a sample with a label not containing the required particulars. The retail sale, however, of a condensed milk with an incorrect equivalent for the pints of milk present is probably no offence, as it is very improbable that the retailer, knowing nothing of its composition, sells it "wilfully," which is necessary for the 1907 Public Health Act.

Section 3. Particulars of compounded drugs have been given in the previous chapter (p. 43). A request for "half and half" coffee and chicory would be a "compound article of food," and also such articles as "milk and coffee tablets," or "rum and coffee."

Section 4. In *Batchelour v. Gee* (1914) one of the Judges said that the scheme of the Act was that a buyer was entitled to get what he asked for, and that if the seller wished to give him something different he should so inform the buyer before the goods are delivered. In *Clifford v. Battley* (1915), on a request for "coffee" a labelled mixture of coffee and chicory was supplied, wrapped up with five other articles. This was considered sufficient though the purchaser did not see the notice at the time of sale, but particular emphasis was laid upon the wrapping at the implied request of the purchaser. The decision might have been different had there been only one article.

An example of fraudulent increase of bulk was the sale of a mixture labelled "chicory and coffee" which contained 74 % of chicory. The vendor was fined as the price was twice as much as it should have been (*Grocer*, 1909, Oct. 23). "Finest essence of COFFEE with chicory" was held to be an adequate defence under this section, unless the mixture was intended fraudulently to increase the bulk, etc. (*B.F.J.*, 1915, 139).

While a label in certain cases is a satisfactory defence, it can hardly be used to *create* an adulteration offence. Suppose coffee were asked for, and the article supplied was labelled "Pure chicory," and that label were accepted as a declaration. If the chicory were adulterated with sand, the vendor could hardly be prosecuted for selling adulterated chicory (as the request was for "coffee"), though he might be for giving a false label.

Section 5. "If any person abstracts," this suggests a physical process, which it may be difficult to prove analytically. Is *addition* of spent ginger to genuine ginger legally *abstraction* of flavouring constituents? The Sale of Milk Regulations authorise the assumption of abstraction when milk is deficient in fat (p. 494).

The rising of cream in a churn is a natural process, and without stirring, the milk drawn from a tap at the bottom will be short of

part of its fat ; the sale of such poor milk is an offence, even if the milk put into the churn was as it came from the cow (*Bridges v. Griffin*, *B.F.J.*, 1925, 53 ; *Thompson v. Nuttall*, *B.F.J.*, 1925, 55 ; *Jones v. Evans*, *B.F.J.*, 1926, 115). In *Sheppard v. Andrews* (1928) the Lord Chief Justice said he could not understand how milk can be said to be in the same condition in which it came from the cow when (1) it is placed in a particular receptacle ; (2) it is left standing about ; (3) it is jolted about in a milk float (*B.F.J.*, 1929, 4, 14, 45).

Sections 17 and 18. The subjects of sampling and of public analyst's certificates have been previously treated (pp. 11, 33).

Section 30. Unfortunately, false labels are not uncommon, and the usefulness of this section is limited by the word " wilful," which is often impossible to prove. In a recent case both retailer and manufacturer were convicted for a false label on bismuth tablets (*B.F.J.*, 1931, 4 ; see also p. 521).

Sections 28 and 31. ANALYST'S CERTIFICATES. The scheme of the Act appears quite simple. It is the duty of the Public Analyst to analyse any sample sent to him, and to certify if it is genuine or otherwise—an impossible thing without standards or limits. If a vendor receive a summons and a copy of the certificate and is satisfied of the accuracy of the latter, he need not require the presence of the analyst ; if he disputes the certificate, he may, and should, require the analyst to be called as a witness. Frequently, however, he thinks his defence is more likely to succeed in the absence of the analyst, gives no notice and disputes the analysis. In such cases the prosecution should ask for an adjournment for the attendance of the analyst to defend his certificate. Finally, if there are conflicting analyses, the Government Chemist may be asked to make an analysis, not to decide the question, but to give the magistrates additional evidence for consideration. It may be noted that the Government Chemist is not a public analyst, and that the Act does not make his certificate evidence in such cases, though it is usually accepted as such. His certificate need not comply with the schedule of the Act, *Foot v. Findlay* (1909 ; *B.F.J.*, 1908, 166). The attendance of the analysts who have signed the certificate may be required for giving evidence.

In spite of the very definite statement of the Act, " the certificate of a public analyst . . . shall be sufficient evidence," magistrates have either refused to accept the uncontradicted evidence of the certificate or the analyst himself, or have shirked their duty to fix a standard, and judge the sample by it. There have been a number of High Court decisions affirming this ; the following are milk cases : *Harrison v. Richards* (1881), *Hewitt v. Taylor* (1896), *Smith v. Kerr* (1896), *Banks v. Wooller* (1900), *Somerset v. Pertwee* (1902), *Elder v. Dryden* (1908), and *Hallimond v. Ketching* (1925).

Two vinegar cases, *Grimble v. Preston* (1914) and *Robinson v. Newman* (1917), dealt with standards for phosphoric acid and acetic acid respectively. The magistrates, who declined to hold that lardine should be free from water, as the only evidence before them of any commercial standard was the composition of samples recently analysed, and dismissed the case, were directed to decide the case, *Rudd v. Skelton Co-operative Society* (1911). Other similar cases were *Roberts v. Leeming* (1905, margarine) and *Preston v. Jackson* (1928, table vinegar) (*Analyst*, 1929, **54**, 32 ; *B.F.J.*, 1928, 114). In the judgment on a meat and malt wine case, the present state of the law was stated. The analyst is entitled to express his opinion as to what the standard of any article should be, in order that the thing sold should not be sold to the prejudice of the purchaser, and if he expresses his opinion in his analysis, the court is bound to accept it unless it is contradicted. Two possible standards were mentioned—(1) a quantitative standard, drawing a hard and fast line ; (2) a minimum standard below which a content of the article should not reasonably fall, *Bowker v. Woodroffe* (1928) (see p. 385).

The Second Schedule entitles a purveyor of milk from whom a sample has been taken to require the local authority to take samples from his wholesale dealer. Unless the purveyor suspects the latter, the provision is not likely to be of much practical use, as he will rarely know that his milk is adulterated before the end of the sixty hours within which the notice must be given. Neglect to give such notice, however, prevents the warranty defence being available (sect. 29 (2) (c)).

STATEMENT OF APPEAL CASES. When there is an appeal to the High Court on a point of law, it is important that there should be proper “ statement of the case.” In *Friend v. Mapp* (1904), the case was so stated that an important question could not be discussed. In *Preston v. Redfern* (1912), the absurd statement by the magistrates that 12 % of extraneous water in milk was due to the absence of stirring, was quoted by the Judges who dismissed the appeal.

OTHER ACTS OF PARLIAMENT. The “ wilful ” labelling offences of the Public Health Act, 1907, have been previously referred to (p. 51). Sect. 6 of the Milk and Dairies (Consolidation) Act, 1915, requires the vendor’s name and address to be on a milk cart or can. Sect. 7 provides that the words “ Machine-skimmed Milk ” or “ Skimmed Milk ” shall be clearly visible on receptacles containing them. Sect. 8 gives power to take milk in course of transit when within the district.

The Milk and Dairies (Amendment) Act, 1922, sect. 4, prohibits the addition to milk, of water, colouring matter, as well as of dried, condensed or reconstituted milk, and the sale of such milk. Under this section a warranty defence is not available, *Reeman v. Knapp* (*B.F.J.*, 1925, 113).

The difficulty in using the Merchandise Marks Act is that Health Committees have no power to incur expenditure under this Act except for foods for which an Order in Council has been made. A vendor who supplied margarine to a Birmingham hospital as Danish butter was prosecuted under this Act, when the Danish Government undertook to pay any costs incurred. A dramatic moment occurred in the hearing when one of the defendant's witnesses confessed that he packed margarine in boxes having the Danish "Lur" trade mark on them.

As the result of adulterated milk being sent to Birmingham, a cowman was sent to prison for two months' hard labour for doing wilful damage to milk by adding to it water from a rain-water tub. Vendors have also been sent to prison for obtaining money under false pretences by selling adulterated articles.

When a wholesale dealer is summoned for "aiding and abetting" a retailer, both can be present at court at one time. In one case in 1927, a farmer was fined £10 for this offence and the retailer £2 for adulteration. Appeal cases on this question are—*Gould & Co. v. Houghton* (1921) and *Bowker v. Premier Drug Co.* (1927). The scope of the offence may be wisely enlarged by adding the words "counselling or procuring," to the charge (p. 381).

When magistrates announce that they have decided to convict a defendant, and the prosecution has called witnesses, an application should be made to the magistrates for "special costs" to cover the expenses of such witnesses. This power is sometimes overlooked.

EVIDENCE

FAIRNESS. Witnesses in food and drug adulteration prosecutions should realise the spirit of the English Law that everyone must be presumed to be innocent till proved guilty. The object of preparing and giving evidence should be to present "the truth, the whole truth, and nothing but the truth" in such a form as to help the magistrates to administer justice. Evidence should be given with fairness, not prejudice; in Birmingham we are justly proud of our tradition in this direction, and on a number of occasions defending solicitors have complimented our witnesses on the fair way in which evidence has been given, and even asked their advice.

The fact that Law, for the protection of the public, requires that vendors shall be responsible for the purity of their wares, produces cases of apparent hardship. Some defendants are obviously innocent of any evil intention, and through carelessness or ignorance have been deceived by others. At the request of the Birmingham magistrates small shopkeepers are informed by the sampling officers as to their rights under the warranty clause. In some of these cases the publicity obtained by a prosecution is the main thing for the

protection of the public, and it is almost a matter of indifference whether a conviction be obtained or not. Sympathetic treatment in such cases will cause a defendant to realise that he is being treated fairly, and get his goodwill not to offend again.

Cases of deliberate fraud, such as watering milk, are very different, and for public protection it is necessary that the amount of the fines inflicted should be sufficient to make adulteration very unprofitable, so that fines should not be considered a negligible part of working expenses. One farmer sending milk to Birmingham who had been successively fined £10, £10, £50 and £160 for milk adulteration, still continued to add water.

“HEARSAY” AND OTHER EVIDENCE. Hearsay must not be given in evidence, except that any statements made by the defendant, or his servants *in his presence*, may be legitimately given, if he has been told that he need not say anything, and that the statements may be used in evidence.

Hearsay may, however, be given if the defence asks for it. An amusing example occurred in Birmingham. An inspector was giving evidence in a case in which a farmer had sent milk low in fat (1·5 % and 1·8 % respectively), and said that the defendant's wife made a statement. At this point he quite correctly stopped. The defendant's counsel thought the statement made would help his case, and pressed the inspector to give it. He was a sadder and a wiser man on hearing that what the defendant's wife had said was : “ We set some of the milk in pans, skim it and mix all the milk together, but I suppose the cook and William omitted to do this the morning you took samples, and put it all in one churn.” The fine was £20. The deficiency of fat in the two churns was equivalent to about 6 lbs. of butter.

Third-party evidence can be secured if necessary by serving a subpœna on a railway guard or porter, or a lorry driver, or a wholesale dealer to prove a contract.

Witnesses must also take care not to prejudice any case by reference to other formal or informal adulterated samples, before the magistrates have decided to convict, except as required by the *defence*, or as rebutting evidence.

In a lime-water case the defendant's solicitor asked me if lime-water did not rapidly deteriorate. I answered that I had reasons for saying that had not happened. On being pressed for the reason I said we had bought a similar defective sample three months before.

REBUTTING EVIDENCE. When a defendant's evidence is to the effect that the adulteration was due to some accident that happened on that occasion only, the purchase of other formal or informal adulterated samples from the vendor may be proved as rebutting evidence. Complete proof should be available though it may not be required. After the magistrates have decided to

convict, an opportunity should be given to acquaint the court as to previous convictions before the fine is stated. A statement (supported by evidence, if required) as to previous formal, or informal, samples may possibly also be admissible.

The plea "guilty" is sometimes made to prevent the magistrates appreciating the amount, or nature, of the adulteration. A prosecuting solicitor should prevent such "smothering" of the case by his opening statement, and by calling evidence, if necessary.

ANALYST'S EVIDENCE. MILK ADULTERATION. If it be possible, a public analyst should attend all adulteration prosecutions, not necessarily to give evidence, but to watch the case on the scientific side, to prompt the prosecuting solicitor, to advise the magistrates on technical points if asked, and generally, to prevent fairy tales being accepted as solid truth.

His evidence should be, as much as possible, provable scientific fact, leaving the bench to make any necessary inference from it. His evidence should be simple, and clearly given, without any display of scientific terms; if that can be avoided.

Cases of adulterated milk sent by farmers will serve as an illustration. Years ago I found that the defending solicitors in such cases would speak lightly of the presence of a pint or two of water in a churn of milk, when gallons had really been added. They either assumed the quantity or presumed the magistrates would not check the calculation. To prevent the bench being thus misled—in every case when there were churns of milk concerned—I calculated the amount of added water, using the composition of the milk taken at the farm by the inspectors as the standard. I rarely, however, said "added water." The usual statement was that, say, 13 gallons of milk of the composition of that obtained at the farm, mixed with 2 gallons of water, would be of the same composition as the 15 gallons of milk for which the farmer was being prosecuted. Or, when fat was deficient, that 15 gallons of the evening milk taken at the farm, after the removal of a pound of butter, would have the same percentage of fat as the milk in question (cp. p. 221). "Added water" was only mentioned when the percentage of solids-not-fat was too low for any possible unwatered milk.

It is often pleaded that a watered milk containing, say, 3.5 % of fat, is above the "standard" of 3.0 %. It should be insisted that 3.0 % is not a "standard," but a presumptive limit. Another similar argument is that the presence of a fair amount of fat disproves the addition of water, but 14 % of water is required to reduce an average milk, containing 3.5 % of fat, to 3.0 % (*B.F.J.*, 1930, 59). The appeal case *Kings v. Merris* (*B.F.J.*, 1920, 88) was against the decision of magistrates that the sale of a milk containing 7.95 % of solids-not-fat was not to the prejudice of the purchaser, as the deficiency of 0.55 % was infinitesimal, and as the purchaser obtained

a better article as the fat was 0.6 % in excess of the limit. The appeal was allowed, and the vendor ultimately fined £1.

In a similar Birmingham case, the defendant's solicitor argued that a deficiency of 0.5 % of solids-not-fat indicated an excess of only 0.5 % of added water—a trivial offence. By the same argument a liquid containing no solids-not-fat has only 8.5 % of added water !

In some cases tables of analyses of the series of milks in question in triplicate (one for defendant) should be prepared. Specimens, or the actual amount of excess water present in a glass of spirits, or even simple experiments, may be shown to help the bench.

Although an analyst has a right to say a food is "injurious to health," it is unwise for him to claim medical knowledge.

Davis v. Blackman (B.F.J., 1924, 115 ; *Analyst*, 1925, 50, 20) is an important case in relation to the admissibility of evidence relating to a considerable number of milk samples taken in the county, to throw light on the question of the genuineness of the milk. The magistrates refused to hear this evidence, but the High Court found it difficult to say such evidence was irrelevant, and that evidence of minima ascertained in similar circumstances was probably of more value than averages. See also *Rudd v. Skelton Co-operative Society* (p. 280). In some cases comparison of the composition of a defendant's article with those sold by other people is most useful. The suggestion that the particular month was a bad one for milk is best answered by giving the average composition of all samples examined during the month, and sometimes it may be added that most of the low samples were obtained from the farm in question.

In all but the very simplest cases the analyst should prepare a proof of his evidence, which helps him to be definite, and he should supply both the solicitor and the sampling officer with a copy of it. This evidence is useful to the solicitor in opening his case. The three officers should be in closest co-operation so that there is no risk of discordant evidence being given in court.

The prosecution has a right to produce the analyst to give evidence, as well as, or instead of, his certificate. He must take care, however, not to suggest other offences besides those contained in the summons.

In cross-examination an analyst should be quite clear in his mind as to how far an "about" in his certificate may reasonably be stretched. Acknowledging ignorance on a particular matter is much wiser than guessing. Once, I was asked if I knew a particular thing, and replied "No." After the case was over the solicitor who asked the question admitted it was a trap, as he had made up the suggested fact. Further, an affirmative answer to a debatable statement might, in an appeal, appear as "the analyst admitted." The witness-box is a very bad place in which to make calculations ; it is much better to offer to give the results later. Some such questions

may be intelligently anticipated, such as, "How much of a particular drug would it be necessary to take to get a minimum dose of arsenic?" Giving opinions in the box on specimens offered by the defence is usually unwise; it is better to say that an analysis is necessary.

Making rash answers, losing one's temper, or asking a question of a solicitor, are all to be avoided, and, on the other hand, the right to give a full answer, and not half one, should be exercised, whether the advocate likes it or not.

OFFICIAL SAMPLER'S EVIDENCE. SAMPLES TAKEN AT FARMS. Many of the previous suggestions are equally applicable here. The officer should prepare a proof of his evidence, and also that of any agent or third party (driver, etc.) (cp. p. 567), and give a copy to the solicitor and one to the analyst. He should also take his sample book to court containing notes made at the time of sale or directly afterwards, and the reserve sample, or what remains of it (p. 27). The importance of accurate observation has been stressed in the chapter on "Sampling" (p. 15). A sampling officer, as prosecutor, has the right to address the Court, and to examine witnesses, in addition to giving evidence himself (*Duncan v. Tims*, 36 L. T. 719).

The most important prosecutions are those for milk from farmers' churns taken in course of delivery (see also p. 22). Evidence should be given as to the time and place of sampling, whether the milk was carried in a milk truck or a guard's van, and that it was thoroughly mixed before sampling. The label on a churn should be produced, as it often bears a guarantee; and it should be stated if the number of gallons indicated on it corresponds with the actual content. The questions of the churn being sealed, and of any numbers on it or on the lid, are sometimes important. Questions may be asked as to the milk travelling without changing, or if transhipped, as to the time it was waiting. Sometimes an officer can give evidence that he has travelled in the same train as the milk, and so absolutely disprove any interference (*B.F.J.*, 1912, 77). Evidence of this kind will prevent a farmer, while admitting his responsibility for the milk, laying the blame on railway servants. Cream, however, may be lost during transit, if the milk churn lid fits badly, or if the churn is carelessly rolled.

The decisions *Hunt v. Richardson* (1916) and *Grigg v. Smith* (1917), that the critical question is "Is the milk as it came from the cow?" attach great importance to the taking of samples at the farm of milk obtained under the sampler's observation. The judgment in *Wilkinson v. Clark* (1916) was that evidence as to the quality of the farm milk was admissible, provided (1) that it was taken from the same cow or herd as the prosecution sample, (2) that it was the same meal, and (3) that it was milked in the same way by

experienced persons on both occasions. It was stated that if a farm sample was defective it could not be used to prove another offence. *Smith v. Philpott* (1920) extended the interval to three days, and further stated the evidence was admissible even if the interval which had elapsed made it worthless.

The position is a curious one. That the conditions of milking were the same on both occasions can only be proved by someone at the farm, and not by a sampling officer. There is, of course, no *right* to go to the farm, though a refusal by the farmer will be suitably assessed by the bench. All a sampler can do is to require the milking to be done by the usual people in the ordinary way, and to trust to the farmer's assurance that it has been done, or if not, what difference has been made. In fairness to the farmer, information obtained at the farm should be given in evidence.

FARMER'S EVIDENCE. The obligation on the defendant is to prove that his milk was as it came from the cow. It is not sufficient to prove it was genuine when delivered to the railway—*Andrews v. Lukin* (*Analyst*. 1917, **42**, 379); *Pollock v. Surtees* (*B.F.J.*, 1924, 54, 106); the burden of proof is upon the defendant to trace by credible evidence, and without a lacuna, the history of the milk from the moment at which it was taken from the cow, to the moment at which it was handed to the purchaser—*Davis v. Blackman* (1924); also *Bowen v. Jones* (1917), *Kings v. Merris* (1920), and *Latham v. Newlove* (*B.F.J.*, 1925, 13, 39).

When a farmer assays to do this a frequent weak spot in the evidence is the protection of the milk during the night. It is sometimes left covered in the open air, or in an unlocked shed, or if locked, the key is left in a place to which several persons have access. If there is a disproportion between the quantity of milk obtained when the inspectors were there and that previously sent, he should be asked to explain the difference. Questions may be asked as to additional sale of milk in the neighbourhood or to any other wholesaler, and as to the quantity of milk fed to calves, or used by his family. If another person takes the milk to the station, the possibilities of sale of milk by the way and addition of water should be investigated.

Should a farmer suggest that water has been added on the railway he may be asked to explain how the thief could possibly go to a station without detection, taking two buckets, one empty to receive the (say) 2 gallons of stolen milk, and the other having 2 gallons of water to replace it! He might also be asked if he complained to the stationmaster, and if not, how it was that the milk improved as soon as the farmer knew of the sampling. Another question might be, "Is your milk so renowned for quality that it will be stolen on several occasions, while other farmers' milk is also available?" Also, why did not he protect his milk by using a lead seal, which is

permitted by the Railway Companies if the "tare" is conspicuously marked on the outside of the churn (*B.F.J.*, 1900, 223).

Sometimes a farmer, producing a little milk, arranges with another to put his milk in the same churn, and thus may be responsible for another man's adulteration.

Cowmen, from laziness in milking, or to boast of the yield, have added water to keep up or increase the apparent production of milk (*B.F.J.*, 1929, 38 ; 1930, 40). In some cases where a gentleman farmer has little or nothing to do with the milking, the question may be asked as to who profits by the adulteration. In cases where fat is deficient the presence of a separator at the farm, or the making of butter, should be enquired into. The sampler who has visited the farm should be in a position to check some of the replies, and also to suggest relevant questions.

A conviction must follow if the vendor fails to discharge the onus of showing the milk was in the same condition as it came from the cows (*Watson v. Skelton*, *B.F.J.*, 1922, 33), and further, the proof must be satisfying, and leave no doubt in the mind of the court (*Walshaw v. Juddick*, *B.F.J.*, 1921, 35, 49 ; *Jones v. Jones* *B.F.J.*, 1924, 65). If the magistrates find the milk is not of the nature, or of the substance or of the quality of the article contracted to be sold, they must convict (*Marshall v. Skett*, 1912) unless they consider the offence trivial, when they may dismiss the summons under the Probation of Offenders Act (*Preston v. Redfern*, *B.F.J.*, 1912, 91).

CHAPTER VI

GENERAL METHODS OF ANALYSIS

Specific gravity. Water. Capacity of filters. Fibre. Nitrogen. Acidimetry. Indicators. Determination of pH.

METHODS for the determination of mineral matter, preservatives, sugars, oils, fats and waxes, and alcoholic liquids are given in later chapters.

Methods for the determination of nitrogen, phosphates, potash, oil, fibre, sugar, etc., are given in the Fertilisers and Feeding Stuffs Regulations, 1928. For the determination of oils in seeds, see the Report to the Ministry of Health (*Analyst*, 1920, **45**, 279). Clacher has described and figured an extraction apparatus (*S.P.A.*, 1910, **35**, 349), as also has Phillips (*S.P.A.*, 1916, **41**, 122), and Self and Corfield (*Q.J.P.*, 1930, 408). The methods given in the B.P. should be consulted, and also those in the official Fertilisers and Feeding Stuffs Regulations, 1928.

SPECIFIC GRAVITY

Wright has given factors for the correction of sp. gr. of oils to 15.5° C. (*J.S.C.I.*, 1907, 513). Siebold found that the sp. gr. of turbid liquids could be taken correctly with an hydrometer if the solid did not deposit too quickly (*Analyst*, 1879, **4**, 189). The application of the Westphal balance to waxes is given later (p. 285). For the determination of the volume of a loaf of bread, the use of a suitable beaker and lentils was found satisfactory. The lentils were shaken down, struck off level, and their volume determined, with and without the loaf in the container. Rice in grains was not quite so good.

CAPACITY OF FILTERS

The following table gives the capacity of folded filter paper on the assumptions that the level of liquid is 5 mm. below the edge of the paper, and that 100 sq. cm. require 1 ml. of water to wet it :—

CAPACITY OF FILTERS										
Diameter (cm.)	7	9	11	12½	15	18½	24	27	32	38½
Capacity (ml.)	7.5	17	33	51	91	180	403	579	983	1735

This table is particularly useful as a guide when filtration is slow.

WATER

For drying all kinds of foods and drugs, the writer has used metal dishes 3 inches in diameter, with straight sides ½ inch high.

German silver, nickel, or aluminium bronze are suitable materials, but must not be used for ignitions. With syrup, honey, etc., a few drops of water should be added, so as to spread out the substance and utilise the whole of the drying surface. If used for evaporation of 50 ml. of a liquid, the sides should be 1 inch high. Stokes has recommended the use of dishes made of stainless steel (*S.P.A.*, 1929, **54**, 538).

The drying of sugar is very tedious; 0.5 gm. of sucrose after wetting was not completely dried at the end of forty-four hours in the water oven. A smaller quantity should be taken, and the drying facilitated by evaporating purified methylated spirit on it. In the presence of acid the drying may be complicated by increase of weight due to formation of invert sugar. When 5 ml. of a 10 % solution of sucrose containing only 0.01 % of citric acid was evaporated down, without drying, one-third of the sucrose was inverted.

Drying on paper coils has been advocated by Graftian (*Analyst*, 1895, **20**, 251) for honey, and by C. C. Roberts for molasses (*Analyst*, 1912, **37**, 560). Another method for such substances is distillation with a volatile liquid and measurement of the water coming over. Xylene was used by van der Linden (*Analyst*, 1918, **43**, 221), and toluene by Jones and McLachlan (*S.P.A.*, 1927, **52**, 383). Middleton described an improved apparatus at the British Pharmaceutical Conference in 1931 (*Q.J.P.*, 474). Calcium carbide was used by Cripps and Brown for the determination of moisture in spices (*S.P.A.*, 1909, **34**, 519), and for infants' foods, etc., by McNeil (*Analyst*, 1912, **37**, 475). About 582 ml. of acetylene are yielded by 1 gm. of water; see also West (*Analyst*, 1916, **41**, 186).

Huntley and Coste have reported on methods of determination (*J.S.C.I.*, 1913, 62). Trowbridge has pointed out that moisture can be determined in almost everything at air temperature by drying in a vacuum over sulphuric acid (*Analyst*, 1910, **35**, 22); see also Skertchley (*J.S.C.I.*, 1913, 70). Baking powder must be dried this way, as carbon dioxide would be lost on heating. Reports of committees of analysts have been made on the determination of moisture in oils (*Analyst*, 1920, **45**, 280) and in condensed milk (*S.P.A.*, 1927, **52**, 403).

FIBRE

The writer prefers the following modifications of the Fertilisers and Feeding Stuffs Regulations: (i.) The omission of the extraction with petroleum spirit if the amount of oil is small. (ii.) Using a 400 ml. beaker, in place of a flask, and after boiling putting in a water bath for one hour, covered with a clock glass, then filtering through No. 595 filter paper with gentle suction. (iii.) Transferring the washed fibre, by washing, to a platinum dish, drying to constant weight, then igniting to determine the amount of loss.

NITROGEN

To a weighed quantity of solid, or volume of liquid, in a round-bottom flask, add 25 ml. strong pure sulphuric acid, about 10 gm. of sodium sulphate, and a small crystal of copper sulphate, and heat in fume chamber. It may be necessary to heat gently at first to avoid frothing. Liquids may be concentrated previous to addition of acid, but care must be taken to avoid charring.

The flask should be lightly closed with a balloon stopper, and the liquid boiled until it is colourless or pale blue. In some cases a further addition of sulphuric acid may be necessary, as no part of the liquid must become dry, or ammonium sulphate may be lost. After cooling, the mass is washed out into a 2-litre distillation flask with 100 ml. water, and 100 ml. of 10*N*. NaOH added, making the liquid alkaline.

A spray trap is attached to the side tube, and the liquid distilled with steam, the flask also being heated if necessary. A tube with a bulb is attached to the condenser, the lower part of it dipping into a measured quantity of *N*/10 HCl, tinted with methyl red, contained in a flask.

About 200 ml. of liquid are distilled, and the distillate titrated back with *N*/10 NaOH. To ascertain that all the ammonia has come over, 100 ml. more are distilled into an empty receiver, and titrated.

A blank should be determined by heating 25 ml. (or more if used) of sulphuric acid with sugar and distilling in the same way as the actual determination.

The efficiency of the spray trap should be proved; violent distillation may carry over alkaline spray. As the ammonia comes over rapidly at first, the rate of distillation should be slow at first, and a piece of red litmus paper should be put in the upper part of the receiving flask to prove that there is no loss of ammonia.

If nitric nitrogen is also required reference should be made to the methods given in the Fertilisers and Feeding Stuffs Regulations.

ACIDIMETRY

According to the electrolytic dissociation theory of Arrhenius, an aqueous solution of an acid is partly composed of the acid, and partly of the products of its electrolytic dissociation. Hydrochloric acid, for instance, is partly HCl and partly H^+ and Cl^- in equivalent proportions. Alkalies in solution are similarly dissociated, sodium hydroxide in solution being partly NaOH and partly Na^+ and OH^- . The acidity and alkalinity of solutions, at a particular time, are proportional to the amounts of hydrogen ion (H^+) and hydroxyl ion (OH^-) respectively, present, while other properties of the liquid, such as the charring effect of H_2SO_4 , depend on the undissociated part.

When solutions of acids and alkalies are mixed, the H^+ and the OH^- combine to form water, and further dissociation occurs, till at the point of neutralisation, no undissociated acid or alkali is present, and H^+ and OH^- are in the minute equivalent proportions in which they occur in water.

A normal solution of an acid contains its equivalent weight in gm. per litre, and if it were *completely* ionised, 1 gm. of H^+ . With the same qualification, a N/100 acid would have 1/100 gm. of H^+ per litre, a quantity which may be expressed as 1.0×10^{-2} . The logarithm made positive (2.0) is the "exponent of hydrogen ion concentration," and is indicated by "pH." As a matter of fact, only about 98 % of the HCl is dissociated in a N/100 solution, the actual hydrogen ion strength being 0.98×10^{-2} , and the pH being 2.00—Log. of 0.98, equal 2.01.

Strong acids are those, like HCl, which have a large percentage of dissociation at moderate dilution. Acetic acid is a weak one, there being only about 4 % of dissociation in a N/100 solution. Its pH, at this dilution, is 2.00—Log. of 0.04, and equals 3.40.

There is a similar difference in alkalies; in N/100 solution about 90 % of NaOH is dissociated, but only about 4 % of the weak alkali, NH_4OH . In all cases the percentage of ionisation is increased by dilution.

The pH of pure water at 15° C. is 7.2 (Noyes, *J.S.C.I.*, 1912, 1013); if the pH of a solution is less than that figure, the solution is acid, and if greater, alkaline.

The "acidity" of a liquid may be one of two different things, either its actual acidity (H^+), which will include the effect of any carbonic acid present; or its power to neutralise alkalies, carbonic acid frequently being ignored. Similarly, "alkalinity" may indicate OH^- only, or the total power to neutralise acids. Further "neutrality" is not confined to pH 7.2, but is often qualified by the indicator used, neutrality to methyl red often being very different from neutrality to phenol phthalein.

For a general discussion of pH, its determination and applications, see Evers (*P.J.*, 1922, March 4, and *S.P.A.*, 1921, **46**, 393), and Lizius and Evers (*S.P.A.*, 1922, **47**, 331).

The most important operation in acidimetry is the preparation of normal HCl. It is not only the standard for acidity, but is also responsible for the standard alkaline solutions. The writer would like to stress the importance of having two *independent* acids in a laboratory. For instance, if an assistant found a vinegar to be weak, the writer would confirm the figure, not only by titration with N/2.NaOH, but also by ascertaining the accuracy of that solution by comparison with N.HCl, which he had prepared and kept for the purpose. N.HCl, kept in a stoppered bottle tied over with washleather, will keep for years with very little change. The

importance of an independent check is not always realised. It is quite easy to fill up a burette containing N/10 with N/2, and return the mixture remaining in the burette to one of the stock bottles. In a case which came under the writer's notice, something of this kind was suspected. Two analysts agreed as to the composition of a sample, but the third differed considerably. When the third analyst was challenged, he replied that he had carefully *checked the calculations* of his assistants !

As strong HCl of the B.P. is 10E, approximately normal acid may be made by diluting 100 ml. to 1,000 ml. Standard HCl may be tested by precipitation with AgNO_3 and weighing AgCl , but as the liquid is used for alkalies, the writer prefers to standardise with an alkali, pure sodium carbonate. This may be prepared by removing any impurity from NaHCO_3 by percolating with water in a filter paper in a funnel, drying and heating in a platinum crucible, half full of the salt, rapidly to 270°C. , and heating for half an hour without exceeding 300°C. (Lunge, *Analyst*, 1903, **28**, 307). Care must, of course, be taken that sulphur is not absorbed during heating.

The pure Na_2CO_3 is put in a weighing bottle while warm. When cold, the bottle is weighed, about 5.4–5.5 gm. put into a beaker, the exact weight being ascertained by difference. Water is added, and 100 ml. of the acid is carefully pipetted into the beaker, after boiling the titration is completed, using azolitmin or methyl red as indicator, care being taken that CO_2 is removed. The determination is repeated, and if the result agrees with the first one, the remaining acid is diluted to normal strength. A third determination is advisable. The strength of the solutions titrated should not be less than N/5 (Lunge, *op. cit.*). Normal acid is diluted to N/2 or N/10, which are more useful strengths.

When several samples of an article have to be analysed, instead of taking an exact amount in gm., time is saved by weighing quantities which are a little less than will be neutralised by, say, 30 ml. of the standard acid, then adding that quantity, boiling if necessary, and titrating back with standard alkali. In other cases taking a weight bearing a simple relation to the molecular weight of the article will simplify calculation. The quantities taken depend on the degree of accuracy required. If about 30 ml. of standard acid be used, each 0.1 ml. is equal to about 0.3 % in a practically pure substance.

In the titration of liquids containing ammonia, loss is avoided by adding the ammoniacal liquid to part of the standard acid, diluted with water, and then completing the titration.

N/2 and N/10 NaOH solutions are made by diluting a strong solution with boiled distilled water. The strong solution is conveniently made by adding 1 lb. pure NaHO to 600 ml. water,

shaking with care, and syphoning from the deposited Na_2CO_3 after standing; it is about 15 E.

The diluted liquids are standardised with N/2 or N/10 HCl, using both methyl red and phenol phthalein as indicators; there should be little difference. In a titration the figure appropriate to the indicator should be used. Standard alkaline solutions should be kept in resistance glass bottles, or deposits occur, due to the action of the alkali on the glass.

The presence of iron salts interferes with the determination of acidity by NaHO, but not when Kiefer solution is used. It is prepared by dissolving 60 gm. of copper sulphate crystals in 500 ml. of water, adding 500 ml. 2E.AmHO, allowing to stand, and if necessary filtering through glass wool. It is about normal; the exact strength is determined by titrating into standard acid and water, the appearance of a permanent precipitate indicating neutrality, no indicator being used.

The detection and determination of free mineral acid in the presence of organic acid is dealt with later, under vinegar (p. 394).

INDICATORS

Indicators are various compounds, one ion of which is coloured, but which when non-ionised are either colourless (phenol phthalein), or have a different colour from the ion (methyl orange). There is a considerable difference in the pH of solutions in the point at which the colour change takes place.

The following table, based on experiments made by Miss E. M. Milward, in the writer's laboratory, gives the colour changes of a selection of indicators and their useful pH range :—

pH RANGE OF INDICATORS

INDICATOR	pH RANGE	ACID TO ALKALI
Thymol blue (1st change) .	1.2- 2.8	Pink—yellow
Benzyl-aniline-azo-benzene sulphonic acid . . .	2.0- 3.0	Reddish pink—yellow
Bromo-phenol blue . . .	3.0- 4.0	Yellow—colourless—blue
B.D.H. “ 4.5 ” . . .	3.5- 6.0	Orange pink—grey—blue
B.D.H. “ Universal ” . . .	3.5-10.0	Red — yellow — green — blue — violet
Methyl orange . . .	3.7- 4.5	Reddish orange—yellow
Methyl red . . .	4.5- 6.0	Red—yellow
“ Sofnol, No. 1 ” . . .	4.7- 6.4	Pink—pale yellow
Bromo-cresol-purple . . .	5.0- 6.5	Yellow—purple
Bromo-thymol blue . . .	6.0- 8.0	Yellow—blue
Cresol red . . .	7.2- 8.8	Yellow—purple
Naphthol phthalein . . .	7.3- 8.7	Pink—colourless—greenish blue
Phenol red . . .	7.5- 8.0	Yellow—purplish pink
Thymol blue (2nd change). . .	8.0- 9.3	Yellow—blue
Thymol violet . . .	9.0-13.0	Yellow—blue

From the above table one or more indicators can be selected for any particular pH required. For determination of neutralising power, a much smaller variety is required, and the requirements are different. Gradual change of colour is an advantage in an indicator for pH, but for showing when neutralisation has been reached, the sharper the colour change, the better. All indicators have some intermediate transition colour, and frequently the neutrality point is not midway between the beginning and end of the change. Methyl orange, for instance, on addition of alkali changes from red to yellow, through orange, but its neutral point is when orange changes to yellow. In the following table, the neutral point is at the beginning or the end of the colour given. The sensitiveness of an indicator may be measured by the quantity of acid or alkali used between the beginning and end of the colour change; in the table they are given as percentages, the total quantity of acid or alkali used being taken as 100. In the actual titrations about 20 ml. of N/10 acid or alkali were used. The percentages depend on the conditions of the experiment, but the figures for the different indicators are comparable for each combination of acid and alkali. Methyl orange, for instance, with 13 %, is less sensitive than the other indicators (1–2 %), with HCl and NaHO. The table depends on experiments made in the writer's laboratory by H. H. Bagnall :—

SENSITIVENESS OF INDICATORS

	Bromo-phenol blue.	Methyl orange.	Methyl red.	Azo-litmin.	Universal (B.D.H.)	Phenol phthalein.	Phenol red.
Neutral colour	blue	yellow	red	red	pink-yellow blue-violet	colour- less	yellow
NaHO and HCl, Colour range %	2	13	2	2	1	2	1
AmHO and HCl, Colour range %	2	5	1	30 ?	—	50 ?	10
H ₂ C ₄ H ₄ O ₆ and NaHO Colour range %	60 ?	60 ?	15 ?	10 ?	1	1	1
Na ₂ CO ₃ and HCl (cold), Colour range %	3	8	30 ?	60 ?	1	30 ?	1
Effect of CO ₂	0.5	1.1	0.5	0.8	0.3	52	50
H ₃ PO ₄ and NaHO, 1st H ⁺ neutral ?	Yes	Yes	Yes	No	Yes	No	No
2nd H ⁺ neutral ?	No	No	No	No	Yes	Yes	Yes
3rd H ⁺ and CaCl ₂ neutral ?	No	No	No	No	Yes	Yes	Yes

It is obvious from the above table that the indicator must be selected according to the strength of the acid and alkali used. Methyl red, though the best indicator for ammonia, is almost useless for tartaric acid; the “?” marks the indefiniteness of a reaction. On the other hand, phenol phthalein, though excellent

for tartaric acid, is useless for ammonia. Methyl orange is often considered to be insensitive to CO_2 , but in the conditions of the experiment, it introduced an error of 1.1 %, methyl red was less affected ; but for precise work, it is better to boil off the CO_2 with excess of acid, and titrate back, previously cooling if methyl orange is the indicator. According to the above results, phenol red appears to be superior to phenol phthalein ; the 50 % effect of the CO_2 shows that phenol red is neutral to NaHCO_3 .

Phosphoric acid being tri-basic, is neutralised in three stages, and methyl orange is the best indicator for the first stage ; it suffers from the disadvantage that in a solution that is slightly acid, it appears alkaline on heating, and has to be cooled before the titration can be completed. The presence of CaCl_2 is necessary for indication of the neutrality of the third H. Further particulars will be found under the heading "Phosphates." Borates, also, have been dealt with under that heading.

For coloured solutions the use of fluorescein is sometimes useful ; for stout, and similar dark liquids, see Lizius (*S.P.A.*, 1921, **46**, 194). Recently the use of ultra-violet light, with quinine or umbelliferone as indicator, has been advocated for such solutions (*P.J.*, 1930, 265). Van Urk has described an universal indicator (*Analyst*, 1929, **54**, 254).

DETERMINATION OF pH

The pH of pure water is very sensitive to traces of acid or alkali, but the addition of salts such as alkali phosphates, citrates, or borates, known as "buffers," much diminishes this sensitiveness.

The following method is a convenient one, using only one quantity of buffer solution and giving a wide range of pH. The water used for the solutions should be good distilled water, reduced by boiling to one-third, and kept in resistance glass containers (*Analyst*, 1927, **52**, 492) :—

Use 50 ml. Nessler cylinders of colourless glass, marked at 15 ml. Wash out before use with tap water and rinse with the liquid to be examined or buffer solution.

Add about 0.2 ml. of B.D.H. universal indicator to about 15 ml. of the liquid and the colour will indicate approximately the pH.

Take 15 ml. of the buffer solution prepared by dissolving the contents of a tube (about 13.3 gm.) of B.D.H. universal buffer mixture (Prideaux's formula, *J.C.S.*, 1924, Vol. 125) in 1000 ml. water. Add a definite quantity of a suitable indicator, and add N/4 NaOH from a burette until the colour matches that of the same amount of the liquid in another cylinder, with the same amount of indicator. The NaOH solution should not be left in the burette.

$$\text{pH} = 3.1 + \text{no. ml. N/4 NaOH used.}$$

For other buffer solutions and tables, see Evers (*S.P.A.*, 1921, **46**, 393, and McIlvaine (*J. Biol. Chem.*, 1921, 183 ; Abstract, *Analyst*, 1922, **47**, 89).

An account of electrical methods for determining pH has been given by Monier-Williams (*S.P.A.*, 1921, **46**, 315) ; see also Considien (*S.P.A.*, 1924, **49**, 332).

CHAPTER VII

NORMAL MINERAL CONSTITUENTS

Total ash. Ash soluble in water. Ash insoluble in acid. Sulphates. Chlorides. Phosphates. Carbonates. Potassium and sodium. Calcium.

IN this chapter no attempt has been made to give a complete account of quantitative methods, but notes on methods particularly suitable for food and drug analysis are given.

TOTAL ASH

What could be simpler than this determination? Take a few grammes of the food in a dish, apply heat with a Bunsen burner, and continue the heat till all the carbon is consumed. If necessary, increase the heat for an hour or two, and weigh the residue. The author must plead guilty to many sins of this nature. The results appear all right unless the determination is repeated. When 3 gm. are taken, or a weight which is not a whole number of grammes, a nice number of decimal figures appears in the calculated percentage of ash; each of them is the "actual result of the experiment" without any "cooking." The terminal figures may be thought to be impressive, but they really only show the lack of arithmetical sense in the operator, as the figures would not be obtained again.

Before an ash is determined there should be consideration of the nature of the food or drug, and also of what is intended to be weighed.

In many cases 5 gm. is a suitable weight to take, and gives a more accurate result than 2 gm. with little more trouble. Generally a luminous argand should be used for ignition. The heat can be easily regulated, and smoking prevents the use of a very high temperature. The application of a gentle heat, and, after fuming has ceased, covering the dish with a strip of platinum, will in many cases yield a satisfactory ash. The application of too great a heat at first will fuse the ash round the carbon and delay complete combustion. Some foods like mustard require a Bunsen flame and are not injured by it.

When KCl or NaCl is present, a gentle heat should be applied till fuming has ceased, then the char should be heated with water and filtered. After the residue in the dish and the filter paper have been dried and ignited, the water extract should be added, the water evaporated, and the dish gently ignited. This method is desirable for milk ashes, about 25 gm. of milk being taken.

Heron has advised the fixation of alkalies as sulphates, and weighing the sulphated ash. After carbonisation, H_2SO_4 is added, but as carbon decomposes the acid, a second or third addition may be necessary. The method was tried for a time on oatmeal, under which figures are given.

The addition of a solution of AmNO_3 to promote the combustion of the carbon has sometimes been advocated, but experiments have shown that the resultant weight may not be the same as that determined without the addition.

When a food, such as ginger, has been adulterated with chalk; or a drug, such as rhubarb, yields it on ignition; the amount of ash is indefinite, depending on the amount of heat used. After the carbon has been burned off, the ash should be allowed to stand with water for half an hour, to dissolve or break down the CaO . Am_2CO_3 solution is then added, the liquid evaporated on the water bath, and the residue dried in the water oven for half an hour. The carbonated ash is weighed without ignition (cp. Liverseege, *P.J.*, 1922, May 20).

A. H. Allen (*S.P.A.*, 1896, **21**, 178) has given a study of the changes that take place in igniting an impure sample of cream of tartar. Roberts has discussed the factors affecting the composition of plant ashes (*S.P.A.*, 1918, **43**, 254).

ASH SOLUBLE IN WATER

As foods and drugs usually contain calcium, their ashes will contain CaCO_3 and CaO , which are sparingly soluble in water. To obtain a fairly definite figure for the amount of ash soluble in water the author suggested the following method (*B.P. Conf.*, 1896, 359): Transfer the ash of 5 gm. to a porcelain dish and add 100 ml. water. Heat to boiling, and keep nearly boiling for five minutes. Filter and wash residue into filter paper with two quantities of about 10 ml. water. Ignite and weigh ash insoluble in water, and subtract from the total ash to obtain the soluble ash. It is sometimes useful to determine the alkalinity of the aqueous solution by titration, using methyl red, or methyl orange if phosphates are present.

The amount of ash soluble in water is not necessarily the same as the ash of a cold water extract, as a soluble organic salt may be made insoluble on ignition, if it contains calcium.

ASH INSOLUBLE IN ACID

When the ash of a food or drug is unusually high, the excess may be due to sandy matter, iron oxide, calcium carbonate, sulphate or phosphate. If the iron oxide be a foreign ingredient which should be weighed with the sand, the ash from 5 gm. should be treated

with 10 ml. cold 3E.HCl for ten minutes, before filtration. Ashes like mustard, which have been fused, are resistant to cold acid, and should be heated on the water bath with 10 ml. 3E.HCl, for half an hour, in a covered dish.

If sand only is required to be weighed, heating with strong HCl may be necessary to remove the iron oxide. In the unusual case of a notable amount of alkaline carbonates being present, silicates may be formed, and evaporation to dryness with HCl, to make them insoluble, may be advisable. The grittiness of the insoluble residue may distinguish between sand and clay.

When a carbonated ash, such as ginger, has been weighed, it may be useful to add a definite quantity of N/2 HCl and allow to stand cold. After filtration and washing, the soluble ash is titrated, and the insoluble ignited and weighed. The soluble ash, after subtraction of the alkalinity, calculated to CaCO_3 , will be unusually high if CaSO_4 was present.

SULPHATES

Ratcliff (*S.P.A.* 1907, **32**, 84) and Cripps and Wright (*S.P.A.*, 1914, **39**, 429) have shown that if acid phosphates are present, as in vinegar and flour, the amount of sulphate in the ash will be low. On the other hand, there is the risk that alkaline ashes may absorb SO_3 from the gas flame. In one experiment 0.5 gm. Na_2CO_3 by igniting in a partly covered platinum dish for two and a half hours gained 0.043 gm. SO_3 .

Experiments by O'Sullivan (*S.P.A.*, 1914, **39**, 425) showed that the sulphates of sodium, potassium and calcium are not appreciably reduced during ignition, but that magnesium sulphate undergoes decomposition unless a carbonate is present.

When small quantities of sulphate are present, as in self-raising flour and impure ginger, the following method has been used: Rub 10 gm. of the material with 200 ml. of 2E.HCl in a mortar. Heat in a beaker on the water oven for six hours, then cool and filter. Wash residue with two successive 50 ml. of E/2.HCl, using no water. Add AmHO (about 70 ml. 6E) till nearly neutral. Heat the solution to boiling and precipitate with 15 ml., or less, of boiling E. BaCl_2 (cp. Elsdon, *S.P.A.*, 1915, **40**, 142; Howard, *Analyst*, 1915, **40**, 461). Special methods are given later for compound liquorice powder (p. 478) and sulphur ointment (p. 533).

The disturbing effects of calcium in the determination of sulphate have been pointed out by van't Kruys (*Analyst*, 1910, **35**, 374), he recommends prolonged heating with acid to remove co-precipitated CaSO_4 . Winkler (*Analyst*, 1921, **46**, 148) states that the determination of SO_4 in water is inaccurate, unless Ca is previously removed as carbonate.

CHLORIDES

O'Sullivan (*loc. cit.*) has shown that chlorides are decomposed on ignition, and in the presence of magnesium sulphate all the chlorine may be lost. Allen and Bodmer have observed (*S.P.A.*, 1878, **3**, 271) that when sodium chloride was evaporated to dryness with a solution of citric acid, 24.5 % of the chlorine was lost, but with acetic acid it showed no loss. Evaporation and ignition with tartaric acid volatilised two-thirds of the chlorine.

Methods for the determination of chlorine have been given by Coste and Shelbourn (*S.P.A.*, 1919, **44**, 162), Drost (*Analyst*, 1925, **50**, 624), and Husband and Godden (*Analyst*, 1927, **52**, 288). For its determination in milk and biological material without ashing see Davies (*S.P.A.*, 1932, **57**, 79).

Chlorides may be determined in beer and other liquids containing organic matter as follows : Add 25 ml. to 0.1 gm. Ca(OH)_2 , containing a known amount of chloride, evaporate and ignite gently till vapour ceases to be given off. Crush the carbonaceous mass in the dish with a pestle, and heat in a covered dish with about 50 ml. of water for half an hour. Filter the liquid into a 100 ml. flask, return filter and residue to the dish, dry and ignite, heat with water, wash into flask, when cold dilute to the mark, shake and let settle. Pipette 50 ml. of the fairly clear liquid into a porcelain dish, add phenol phthalein, and neutralise with N/20 HNO_3 . Add E. K_2CrO_4 , free from chloride, and titrate with N/70.9 AgNO_3 solution. A correction should be made for the amount of silver solution required to turn the indicator about 0.1 ml. If n = ml. solution required by 50 ml. of filtrate, and c = chlorine present in 0.1 gm. Ca(OH)_2 , then grains per gallon of chloride expressed as $\text{NaCl} = 2.31 (2n - c)$. As the end reaction is good, the above strength of silver solution is better than N/10.

An approximate determination may often be made by titrating the neutralised diluted beer directly with the silver solution, using a beer with indicator as colour standard.

PHOSPHATES

When phosphoric acid is titrated with NaHO in the presence of methyl orange, one-third of the acidity is neutralised, when the indicator just turns yellow. For this titration methyl orange has been found to be better than several substitutes that have been tried. When phenol phthalein is used, two-thirds of the acidity is indicated, and if neutral CaCl_2 solution be added, precipitation occurs and the liquid becomes acid. The titration being continued, when addition of more CaCl_2 fails to produce acidity, the reaction is complete and the remaining one-third of the acidity has been neutralised. It follows, therefore, that in the presence of a small

proportion of calcium salts, titration with phenol phthalein is useless unless an excess of CaCl_2 has been added. This reaction is important, not only for the determination of phosphates, but also to separate them from borates.

If HCl is present as well as H_3PO_4 , the first titration will be more than half the second. In the presence of NaHCO_3 , an excess of standard acid should be added, the CO_2 boiled off, and the titration continued as before. The calculation may be made as follows :—

A = ml. $N/10$ NaOH to make neutral to methyl orange.

B = ml. $N/10$ NaOH required between methyl orange and phenol phthalein, using excess of CaCl_2 .

C = ml. $N/10$ HCl used to make neutral to methyl orange.

$N/10$ H_3PO_4 or P_2O_5 = $\frac{3}{2} B$ (or $3A$ if other acid absent).

$N/10$ HCl = $A - \frac{1}{2} B$.

$N/10$ NaOH = $C + \frac{1}{2} B$.

With a pure superphosphate, as $\text{CaH}_4\text{P}_2\text{O}_8$, the amount of NaHCO_3 will be indicated by “ C .”

Sodium pyrophosphate ($\text{Na}_2\text{H}_2\text{P}_2\text{O}_7$) is neutral to methyl orange, and about half its acidity is indicated on titration using phenol phthalein and CaCl_2 . Previous heating with acid is necessary to obtain the full reaction.

Aluminium phosphate (AlPO_4), which may be separated by its insolubility in water or acetic acid, is monobasic when titrated with phenol phthalein and CaCl_2 , 1 ml. N being equivalent to 0.122 AlPO_4 .

Phosphates may be determined in the ash of foods as follows :— Add 10 ml. $10E$. HCl and heat in a covered dish on the water bath for half an hour to convert any pyrophosphate into orthophosphate. Add $3E$. NaOH in excess, acidify with E . HCl , boil five minutes and cool. Add methyl orange and neutralise just to yellow, with $N/10$ NaOH , add 1 ml. of phenol phthalein solution and neutral E . CaCl_2 solution and titrate with $N/10$ NaOH . The titration must be continued until acid is not developed by addition of more CaCl_2 solution. In these conditions phosphoric acid is dibasic.

This method is satisfactory unless too much phosphate is taken, and is often more convenient than the use of molybdenum.

The small quantities of phosphates in foods may be determined by uranium as follows :—

Dissolve the ash in $3E$. HNO_3 , filter to remove traces of carbon, etc., and evaporate to dryness to convert metaphosphate to orthophosphate. Dissolve in $3E$. HNO_3 , neutralise, and make slightly acid with $\text{H}\bar{\text{A}}$. Put the solution in a beaker marked at 75 ml. and add 5 ml. of E . $\text{Na}\bar{\text{A}}$ in E . $\text{H}\bar{\text{A}}$. Titrate with $N/5$ Ur solution, using K_4FeCy_6 powder, or solution, as an external indicator. Heat in water bath and complete the titration after dilution to the

mark. As acetic acid solution of calcium phosphate may precipitate on heating, in the second determination, nearly the whole of the Ur solution should be added before heating. The Ur solution should be standardised in the same conditions as in the determination. The presence of FePO_4 retards the reaction, but the small quantities usually present in foods are determined.

W. Smith read a paper before the British Pharmaceutical Conference (*Q.J.P.*, 1929, 238) on the volumetric determination of phosphoric acid. Neuman's volumetric method (*Analyst*, 1909, **34**, 507) has been commended for foodstuffs (*S.P.A.*, 1911, **36**, 582). Tillmans and Bohrmann have given a method for determining ortho-, meta- and pyro-phosphates in foods (*Analyst*, 1921, **46**, 199).

CARBONATES

DETECTION, RAE'S METHOD (*P.J.*, 1928, Oct. 6). Into a wide-mouth flask put about 1 gm. of the substance, about 25 ml. of water free from CO_2 , 2 ml. of chloroform, and 10 ml. 3E.HCl. Close flask with bung carrying a safety funnel having two bulbs containing a little lime-water. Place the flask on the water bath; if there is no milkiness in two or three minutes, the amount of CO_2 present is less than a milligramme. If sulphites, sulphides or nitrites are present, add an excess of 1 w/v KMnO_4 solution to the flask. Too long heating may distil the chloroform and produce a milkiness, which, however, is not removed by HCl.

DETERMINATION, MODIFIED HEPBURN'S METHOD (*S.P.A.*, 1926, **51**, 622). A thick-walled conical flask about 1,000 ml. capacity with a neck about $1\frac{1}{2}$ inches in diameter, is closed with a rubber bung through which passes a glass tube, pointed at the lower end, and connected with a piece of thick-walled rubber tubing closed by a screw clip.

The substance to be tested is put in a thick-walled rimless test tube, about $1\frac{1}{4}$ inches diameter, and long enough to reach to within $1\frac{1}{2}$ inches of the top of the flask. About 0.5 gm. is suitable for baking powder, and about 5 gm. for self-raising flour. For dyes and substances which froth badly, Callan recommends wetting with 1–2 ml. alcohol (*S.P.A.*, 1927, **52**, 222).

Pipette into the flask 25 ml., or 50 ml., of E/10 Ba(OH)_2 , according to the amount of CO_2 expected to be present, insert the test tube containing the substance, close the flask with the bung and exhaust with the water pump till bubbles of air are seen rising from the baryta solution. The screw clip is then closed and a separating funnel inserted in the thick rubber tubing. By cautiously opening the screw clip and the tap, a sufficient quantity of water or 3E.HCl is gradually drawn into the test tube from the separating funnel, the flask being shaken. The screw clip is then closed, and a

separating funnel containing water is left in place, so that water, and not air, shall be drawn in, if there be leakage.

After occasional shaking the flask is allowed to stand all night, or longer, if the powder be not wetted throughout. The test tube is removed and the liquid titrated with N/10 HCl, using phenol phthalein, with as little exposure to air as possible. The difference between this figure and that previously obtained by titrating the quantity of baryta solution used, when multiplied by 0.0022 gives the amount of CO₂ present.

In these conditions, theoretical results are obtained and bicarbonates do not yield CO₂; also, the NH₃ in ammonium carbonate does not interfere if NaHO is added before exhaustion; any NH₃ given off is absorbed by the added acid, and does not interfere with the baryta titration.

OTHER METHODS. Macara has described a process for determining carbon dioxide in baking powder, etc., by boiling it out and absorbing with baryta water (*S.P.A.*, 1904, **29**, 152, and 1915, **40**, 272). Dunncliff determines the carbonates in highly coloured liquids by measuring the CO₂ evolved (*S.P.A.*, 1924, **49**, 426). For the separation of carbon dioxide and volatile acids, Foreman (*Analyst*, 1928, **53**, 299) has shown that air passed through a solution of them in 87 % alcohol, well below 85° C. will remove the carbon dioxide only.

POTASSIUM AND SODIUM

After the removal from an ash of sulphates by BaCl₂, phosphates and magnesium salts by pure Ca(OH)₂, and barium and calcium salts by Am₂CO₃ and pure Am₂Ox, the filtrate is evaporated to dryness, and gently ignited to remove AmCl. A little water and Am₂Ox are added to the residue, and after standing all night, filtered to remove traces of insoluble matter and calcium. The filtrate is acidified with HCl evaporated to dryness in a weighed dish, gently ignited, and the mixed chlorides weighed. An excess of PtCl₄ is added in excess to the solution of the chlorides, and after evaporation nearly to dryness on a water bath that is not boiling, the sodium salt is dissolved out by alcohol S.G. 0.864. Dehydration of the platinum salts interferes with solution by the alcohol. The filtrate is evaporated to dryness with H₂SO₄ and ignited. After the reduced platinum has been filtered out, the filtrate is evaporated to dryness, ignited and the Na₂SO₄ weighed. The filter paper containing the K₂PtCl₆ is opened out and exposed to the air till all the alcohol has evaporated, then treated with a little boiling water and the filtrate evaporated to dryness in a weighed dish, and dried in the water oven. The writer prefers this procedure to the use of tared filter papers. The sum of the NaCl and the KCl calculated from these

determinations should closely approximate to the weight of the mixed chlorides previously determined.

Spencer and Sen (*S.P.A.*, 1929, **54**, 224) have claimed that increased accuracy may be obtained by using bromides instead of chlorides.

CALCIUM

To determine calcium in the presence of iron and phosphate :—
Dissolve the ash in dilute HCl and filter. Neutralise with AmOH , boil, add H_2Ox , which dissolves the turbidity, then add Am_2Ox , allow to stand, filter, etc.

Evers has discussed the conditions for the detection of small quantities in drugs (*S.P.A.*, 1931, **56**, 293).

CHAPTER VIII

COLOURING MATTERS. METALLIC IMPURITIES

Colouring matters. Arsenic, determination in beer, flour, apples, vinegar. Lead, determination in Glauber's salt, cream of tartar, self-raising flour, beer. Copper, detection and determination in peas. Tin, canned foods, determination, prosecutions. Zinc. Alum, detection and determination in bread, baking powder.

COLOURING MATTERS

THE question of coloured foods has been much simplified by the issue of the Public Health (Preservatives, etc., in Food) Regulations, 1925. Besides metallic colours, they prohibit the use of six yellow dyes—gamboge, and five coal tar colours, some of which are obsolete in commerce. The following is a simple scheme for their detection :—Heat the coloured food with water and sodium sulphite slightly acidified with HCl, and a piece of nun's veiling. If the fabric is dyed, squeeze out the moisture, and observe the reactions obtained with parts of it and the following solutions :—

Dye	3E.HCl	E/2.NaOH	SnCl ₂ +HCl	KCN
Gamboge . . .	—	Brown . . .	Decolourised	0
Victoria yellow . . .	Paler yellow	Darker yellow . . .	„	Red
Manchester yellow.	„	„	„	„
Aurantia . . .	„	Little change . . .	0	0
Aurin . . .	Brown . . .	Pink . . .	0	0
Picric acid . . .	Pale yellow . . .	{ Brown, or darker yellow }	Decolourised	Red

Gamboge does not dye a fabric in an acid solution.

Nicholls makes an ammoniacal extract of the coloured food, and extracts the dye from the liquid with ether. He gives a scheme for the differentiation of the prohibited dyes (*S.P.A.*, 1927, **52**, 585; 1929, **54**, 335). Other methods are given by Jamieson and Keyworth (*S.P.A.*, 1928, **53**, 418). Chapman and Siebold have given particulars of the absorption of some dyes (*S.P.A.*, 1912, **37**, 339), and Soep of the detection of sandal-wood colouring (*S.P.A.*, 1927, **52**, 696). Drake-Law has considered the whole question, and a discussion followed his paper (*J.S.C.I.*, 1926, XLV., 428T).

A Joint Committee has considered the amount of arsenic that may be allowed in food colouring matters, and recommended limits of 1·4–5 parts per million (*S.P.A.*, 1928, **53**, 217). Methods of determination were given later (*S.P.A.*, 1930, **55**, 102).

The use of ferruginous colouring matter carries with it the risk of arsenic; Armenian bole, for instance, may contain 800 parts per million (*Analyst*, 1926, **51**, 413).

ARSENIC

Before 1900 little attention was paid by analysts to the presence of arsenic in foods. In that year there was an important object-lesson on the risks consumers may run through the carelessness of producers.

Severe epidemics of peripheral neuritis in Lancashire and Staffordshire were found to be due to arsenical poisoning, and a Royal Commission was appointed to investigate the matter. The Commission sat for three years and reported that at least 6,000 cases of illness and seventy deaths had occurred through the presence of arsenic in beer (Final Report, p. 5).

The breweries concerned had bought glucose or invert sugar from one manufacturer who had for about nine months been using highly arsenical sulphuric acid in preparing them. For many years pure sulphuric acid had been supplied, but owing to carelessness or misunderstanding at the acid works, commercial sulphuric acid prepared from pyrites had been substituted. The tragedy which followed was due to the neglect of the brewers, and the brewing sugar manufacturers, to have any analyses made to ascertain if the materials used were suitable for food.

The sulphuric acid used contained 1.4 % of arsenic, or even 2.6 % (Campbell Brown, Report, II., 230), and some of the brewing sugars contained 5 to 9 grains of arsenic per lb. (Report, II., 201, 235). Some of the beer contained 1/10 grain of arsenic per gallon, and in a few cases 1 grain. The manufacturers had prepared a table syrup which contained 1 grain of arsenic per lb. with the impure acid, but fortunately none of it had been sold.

According to the L.G.B. Reports for 1900-1, 7.1 % of the samples of beer examined under the Sales of Food and Drugs Acts were condemned for the presence of arsenic. A number of vendors were prosecuted and fined.

In 1902 there was a minor epidemic of arsenical poisoning by beer which was traced to malt which had been dried with arsenical gas coke instead of anthracite. Some of this beer contained 1/16 grain of arsenic per gallon, and some malt used 1/30 grain per lb. (Final Report, p. 9). Particulars of arsenic estimations relating to malt-kilns have been given by Fairley (*S.P.A.*, 1901, **26**, 177).

The Commission published three volumes of report and evidence, and concluded that prosecutions might properly be instituted for the sale of any liquid food, or liquid entering into the composition of food, which contained 1/100 grain of arsenic per gallon, or of any solid food, whether taken by itself or after mixing with water, if it contained 1/100 grain of arsenic per lb. (Final Report, p. 50 ; also *Analyst*, 1904, **29**, 66). These proportions are still used as a working standard.

In 1907 a report by MacFadden to the Local Government Board recommended the application of the 1/100 of a grain of arsenic per lb. limit to tartaric acid, cream of tartar and citric acid (L.G.B. Food Report, No. 2). There had been prosecutions for the two former in 1904.

In 1920 the carriage in a railway van of a barrel of sugar by the side of a leaky drum of weed killer caused sixty persons who had eaten the sugar to suffer from arsenical poisoning (*B.F.J.*, 1920, 36).

In 1916 the L.G.B. sent out a circular calling attention to the presence of arsenic in baking powder due to the use of impure acid calcium phosphate (*B.F.J.*, 1917, 204). In 1925 a similar circular was issued by the Ministry of Health in relation to the importation of apples on the skin of which arsenic was present.

The B.P. of 1898 prescribed no test for arsenic in sodium phosphate, and great interest was taken when, in 1900, a prosecution was instituted for the sale of effervescent sodium phosphate containing arsenic. Two years later a chemist was fined for the sale of glycerin which contained only 1 part of arsenic in two millions. The editorial of a pharmaceutical paper described the case as "A miscarriage of justice." A report by Campbell Brown on arsenic in glycerin is included in the Report of the Arsenic Commission (II., 233, 255).

Particulars of the prosecutions for arsenic in the above-mentioned articles, as well as in bread, cocoa, confectionery, borax, reduced iron, Epsom salt, milk of sulphur, salt of tartar and bicarbonate of soda, are given under the names of the various articles in the following pages.

In connection with the revision of the B.P. a report was made by Dunstan and Robinson on the determination of arsenic in drugs (*Analyst*, 1904, 29, 375). Hill and Collins described an apparatus for applying the Gutzeit test (*C. and D.*, 1905, Sept. 30). Hill also suggested maximum limits for arsenic in drugs (*Y.B.P.*, 1908, 45). Much of this work was incorporated in the 1914 edition of the B.P. Cocking examined these limits and found no reason for making them less stringent (*Q.J.P.*, 1929, 570).

Voelcker, and also Angell, grew plants on specially arsenicated soil, and failed to find any effect on ripe grain, fruit or seed (Arsenic Commission, Final Report, 34). Jadin and Anstruc found that fresh and dried vegetables and fruits contained 0.2 part of arsenic per million or less (*Analyst*, 1912, 37, 254).

DETERMINATION. Paul and Cownley discussed the qualitative tests for arsenic in food (*P.J.*, 1900, June 30 ; 1901, Feb. 9).

The Manchester Brewers' Association appointed a Commission to examine brewing materials, and Reports issued in 1900-1 (*Analyst*, 1901, 26, 13 ; Arsenic Commission Report, II., 201) recommended

the Reinsch test. Other references to it are—Cowley and Catford (*P.J.*, 1904, Dec. 17), and Evans (*S.P.A.*, 1922, **47**, 6). A research into the theory and practice of the test and its application to As, Sb and Sn has been published by Evans (*S.P.A.*, 1923, **48**, 357, 417).

In 1902 a conjoint Report of the Society of Public Analysts and of the Society of Chemical Industry recommended the use of the Marsh-Berzelius method and gave details for its use and for the preparation of pure reagents for it (Arsenic Commission, II., 206; *S.P.A.*, 1902, **27**, 48, 210). In the same year Thomson published a series of papers criticising the conjoint method. He advocated cooling the deposition tube to give better mirrors, and gave a number of photographs of them (*B.F.J.*, 1902, 170, 191; 1903, 219; 1904, 126, 152, 171). He also used lead kathodes for the electrolytic method (*B.F.J.*, 1906, 166). Papers by Chapman and Law (*S.P.A.*, 1905, **30**, 306; 1906, **31**, 3), Chapman (*S.P.A.*, 1907, **32**, 247) and Evans (*S.P.A.*, 1920, **45**, 8) deal with the insensitiveness of zinc, and the effect of Cd, Cu, Fe and other metals on the evolution of AsH_3 . The latter gave a method for determining arsenic in organic compounds by determining the increase in the weight of Cu.

The Board of Inland Revenue appointed a committee to advise tests for ingredients of beer liable to be contaminated with arsenic. Thorpe gave details of the electrolytic method, using platinum electrodes, which avoided the use of zinc (*S.P.A.*, 1903, **28**, 349; Arsenic Commission, II., 208), and also a method for determining arsenic in fuel (*S.P.A.*, 1903, **28**, 344). Monier-Williams described an improved electrolytic method with lead electrodes, and stated the precautions necessary in the presence of phosphates (*S.P.A.*, 1923, **48**, 262). Evers modified the latter method and applied it to the determination of arsenic in chemicals (B.P. Conf., 1926, 540; Abstract, *Analyst*, 1926, **51**, 526).

Cox (*S.P.A.*, 1925, **50**, 3) has given particulars of the methods used by the Swedish Commission for the determination of small quantities of arsenic.

The author has found the 1914 B.P. Gutzeit process as modified below very satisfactory. The use of strips of mercuric chloride paper increases the range of the test and makes comparison of stains easier. The modification was proposed by Sanger and Black, who gave plates showing the colours of strips with various amounts of arsenic (*J.S.C.I.*, 1907, 1113; *Analyst*, 1908, **33**, 28); also see Lerrigo (*S.P.A.*, 1928, **53**, 90). The B.P. test is given to show that chemicals do not exceed a definite limit of arsenic and the use of hot water, and putting the bottle on a hot plate may save time. The writer considers that when *determinations* are being made, instead of simply testing for *excess*, heat should be avoided as tending to produce irregularity in the evolution of AsH_3 , and

therefore less uniform stains. The use of plumbised cotton wool is also preferred to paper.

The stock standard solution is made by a ten-fold dilution of B.P. Liquor Arsenici Hydrochloricus, which is a 1 w/v solution. 1 ml. of the dilution when made up to 100 ml. gives the B.P. standard, containing 1 of As_2O_3 in 100,000.

The following table gives the amount of arsenic present when various quantities of substance are used and 1 ml. of standard arsenic solution is found to match the stain :—

Gm. or ml. substance taken	1	7	28.35	70	500
Parts As_2O_3 per million	10	1.4	0.35	0.14	0.02
Grain As_2O_3 per gallon	7/10	1/10	1/40	1/100	1/710
Grain As_2O_3 per lb.	1/14	1/100	1/405	1/1000	--

The mercuric chloride papers are made by wetting 11 cm. filter papers with $2\frac{1}{2}\%$ HgCl_2 solution, and drying in the water oven for a few minutes; longer drying will make the paper brittle. The paper is cut into strips about 70×4 mm. and kept in a dark place. Weaver (*S.P.A.*, 1923, **48**, 65) states that damp papers may give no stain.

A 4 oz. wide-mouth bottle is used in which is tightly placed a rubber bung carrying a glass tube about 200 mm. long and 5 mm. internal diameter. The lower end is drawn out to a diameter of 1 mm. and has a hole 2 mm. in diameter at the constriction.

The tube is loosely packed with plumbised cotton wool, leaving a space of about 70 mm. at the top, upon which a strip of HgCl_2 paper is placed. Cotton wool is used which has been saturated with E lead acetate solution, the excess squeezed out, and dried in the water oven. It must be renewed when half of the length is turned black.

The cold solution to be tested is placed in the bottle, and usually about 4 gm. arsenic-free granulated zinc added; the bung is then inserted, and the bottle left in the dark overnight, or longer if the action is incomplete.

The stain may be tested with 10E.HCl. A sulphur stain lasted two hours but was gone in the morning; an arsenic stain lasted all night. Wilkie (*S.P.A.*, 1923, **48**, 64) considers that 10–15% of HBr is the best developer of the stain. Bird (*S.P.A.*, 1901, **26**, 185) gives the effect of HCl on stains produced by SH_2 , PH_3 , SbH_3 and AsH_3 .

Standards are prepared at the same time, and the quantity of material must be adjusted to give a moderate stain, as the graduation of heavy stains is poor.

The B.P. gives directions for the application of the method to drugs, and examples of its use for foods are given below :—

Beer. Add two or three pieces of stick Zn (10–15 gm.) and 5 drops of B.P. AsT. stannous chloride solution to 70 ml. of beer,

and 10 ml. 10E.HCl. Allow to stand two days in the dark. Stick Zn acts more slowly than granulated, and diminishes frothing. Standards are made with 1.0, 1.5, and 2.0 ml. of solution, diluted with mixed beer free from arsenic. A test should also be made of the materials used.

Flour. Elsdon's method detects one part of arsenic per million. Boil 10 gm. with 20 ml. 10E.HCl and 80 ml. water in a porcelain dish for one hour. Cool, put in the bottle, add Zn and allow to stand overnight.

Apples. Weigh one large or two small apples, peel and warm peel with 10 ml. 10E.HCl and 40 ml. water. Cool, decant into bottle, wash by decantation with about 30 ml. water, and add Zn.

Vinegar. Take 50 ml. in a 250 ml. flask, add 5 ml. 10E.HCl, and 7 gm. of Zn, and allow to stand all night.

LEAD

The earliest prosecutions known to the writer for lead in drugs were for cream of tartar. The 1885 B.P. gave no test for lead, but the 1898 edition required that tests for it should "yield no characteristic reaction." A prosecution in 1902 was withdrawn as all cream of tartar was said to contain lead. In 1907, MacFadden, after consultation with manufacturers and dealers, recommended that amounts of lead in cream of tartar, tartaric acid and citric acid below 20 parts per million should not justify their condemnation (L.G.B. Food Report, No. 2, see also discussion on it; *S.P.A.*, 1908, **33**, 174). These limits were incorporated in the 1914 B.P., which also gave lead limits for many other drugs (see Cocking, *Q.J.P.*, 1929, 570). Some of these are too stringent to be used as standards under the Food and Drugs Act, unless the article is definitely asked for as B.P. quality. With these small quantities the experimental error may be appreciable, and as medical evidence on their effects is conflicting, it is probably advisable not to prosecute unless the proportion present is at least twice the B.P. limit for lead.

There have been prosecutions for lead in tartaric acid, citric acid, lime-water, ale, ginger beer, vinegar and confectionery, in addition to some keenly contested ones for cream of tartar. The L.G.B. Report for 1890 mentions Worcester Sauce containing a dangerous amount of lead.

The Preservatives in Food Regulations (1925) prohibited the use in food of any colouring matter containing a compound of lead. Chapman and Linden (*S.P.A.*, 1926, **51**, 563) found 5-25.6 parts of lead per million in dried lobster, mussels, etc., corresponding to about one-fourth as much in the wet edible portions. Methods for the determination of lead in gelatin have been given by Trotman and Sutton (*S.P.A.*, 1924, **49**, 271), and in calcium phosphate by

Warren (*S.P.A.*, 1919, **44**, 199), Miller (*S.P.A.*, 1923, **48**, 263), and Nicholls (*S.P.A.*, 1931, **56**, 594); see also Fox (*S.P.A.*, 1922, **47**, 468). Nakaseko and Nakano (*Analyst*, 1926, **51**, 46) give three methods for the destruction of animal tissues and the determination in them of small amounts of lead.

Richardson has given a method for determination of minute amounts of lead and copper (*S.P.A.*, 1930, **55**, 323).

The determination of traces of lead by H_2S was discussed by Miller (*J. Chem. Soc.*, 1865, 129). Warrington (*J.S.C.I.*, 1893, 97) advised the determination to be made in alkaline solutions, and pointed out the necessity of using for comparison solutions containing the pure substance. Solutions of lead in tartaric acid were darker than similar solutions in water only. C. A. Hill (*C. and D.*, 1905, March 11) showed that the effect on the colour of PbS was the same in solutions containing 4 % and 24 % of a salt, and so the use of a *pure* salt for comparison was not essential. This use of a "dummy" was followed in the 1898 B.P.

Thresh (*S.P.A.*, 1921, **46**, 272) uses $E.H\bar{A}$ containing 0.1 % of gold leaf gelatin. Experiments in the writer's laboratory showed that the addition of gelatin gave colours which were clearer, deeper and more orange, and also more permanent, than with $H\bar{A}$ only. The addition also diminishes the sensitiveness to variation in acidity. The results are equally good in alkaline solutions containing KCN.

Evers (*B.P.C.*, 1920, 405; *Analyst*, **45**, 391) advocates the use of bromo-phenol blue to indicate the correct degree of acidity (about pH 3.8).

When lead is determined in acid solutions the effect of the sulphides of other metals may be serious, and in more than one prosecution there has been a suspicion that all or part of the darkening produced by H_2S has been due to copper and not lead. To prevent this, Teed (*S.P.A.*, 1892, **17**, 142) added a solution of KCN, which prevents the interference of copper and iron by converting them into double cyanides. This method was adopted by the 1914 B.P. Tatlock and Thomson (*S.P.A.*, 1908, **33**, 176) proved that 1 mgm. of either copper, tin, mercury or nickel in the 50 ml. of solution, did not affect the lead result. The addition of hydrogen peroxide will prevent the interference of stannous sulphide.

Andrew (*S.P.A.*, 1924, **49**, 129) found that amounts of iron not in excess of 0.4 mgm. have no effect. The presence of lead should be confirmed by a more characteristic test. 100 ml. of water containing 0.1 mgm. of lead gave a marked cloud in a cylinder on the addition of a small quantity of powdered $K_2Cr_2O_7$; with 0.08 mgm. there was a slight turbidity, but none with 0.02 mgm. Acetic acid should not be added (cp. Allen, *S.P.A.*, 1884, **9**, 195). B. Jones has modified the test for the determination of small quantities (*S.P.A.*, 1930, **55**, 318).

The directions of the 1914 B.P. to filter, before making the lead comparison, ignore the possible presence of metallic lead, or insoluble lead salts, and also the power of cellulose to adsorb lead (see Elsdon, B.P. Conf., 1912, 501, and *P.J.*, Aug. 3).

When the solution of a salt is coloured the standards may be coloured to match with burnt sugar, though the use of Lovibond tintometer glasses over the cylinder containing the standard is more convenient. An alternative method is to match the original colour by adding standard lead solution to the "dummy," and subtracting this correction from the final comparison. When much colour is present C. A. Hill (*op. cit.*) recommends charring, treating with strong HNO_3 and H_2SO_4 , and burning to a white ash. When the ash is very small the addition of a little chalk is advisable. The PbSO_4 is dissolved in solution Am \bar{A} and determined colorimetrically.

When a number of samples of a drug are being tested it is convenient to add H_2S or Na_2S to all, and dilute the palest for use as a "dummy," with correction for its lead if necessary. When in an acid solution the total metal expressed as lead is below the limit, any further refinement of the test is superfluous.

Below are given examples of the application of the above methods to different substances. The cylinders used should be of colourless glass, of similar internal diameter, and ground flat and polished at the bottom :—

Water. *Detection.* To each of two quantities of 200 ml. in cylinders, add 4 ml. gelatine E.H \bar{A} , to one add 10 ml. saturated H_2S solution, mix and compare colours.

Determination (copper being absent). If copper and lead are both present (see Winkler, *Analyst*, 1913, **38**, 123), add acetic acid to water free from lead and adjust colour if necessary, and add lead solution (1 ml. = 0.0001 Pb) before adding H_2S solution. If match is not correct, 0.2 or 0.4 ml. more lead solution may be added to either, as a guide to the quantity of lead solution to be taken for the second comparison. If the lead present exceeds 0.15 per 100,000, it may be advisable to use 100 ml. or 50 ml. for the test. Turbidity in waters may be due to lead oxy-carbonate, and gelatine E.H \bar{A} should be added to the water in the bottle.

Glauber's Salt. Dissolve 20 gm. in about 80 ml. water, filter into 100 ml. flask, if not quite bright, and wash filter to make 100 ml. To 50 ml. add 1 ml. gelatin E.H \bar{A} and 3 ml. saturated H_2S solution. Prepare standards with a 4 w/v solution of sodium sulphate free from lead, or nearly so, to which lead solution (1 ml. = 0.0001 Pb) and the above reagents have been added. The result will be total metals expressed as lead, and if excess be present the determination must be repeated in alkaline solution with KCN. If the liquid has been filtered, treat the filter with 10 ml. of gelatin E.H \bar{A} , and wash to about 50 ml. with water. Add 1 drop of bromophenol blue

solution and lead-free AmHO till the colour just turns blue, then destroy the blue colour by cautious addition of N/10 HCl. Prepare standards with distilled water and 1 drop of bromophenol blue solution, neutralising with gelatine E.HA (about 1 ml.).

Cream of Tartar. To 10 gm. add 2 ml. gelatin E.HA, water, and lead-free AmHO till alkaline, and dilute to 100 ml.; filter if necessary. To 50 ml. add 1 ml. B.P. KCN solution and 2 drops of Na₂S solution. Standards are made with a 4 w/v solution of KHT to which has been added 1 ml. gelatin E.HA, with sufficient AmHO to make it alkaline. If filtration has been necessary, insoluble lead must be tested for as in sodium sulphate.

Self-raising Flour. Ignite 10 gm. in porcelain dish of about 5 cm. diameter. Add 2 ml. 10E.HNO₃, and let stand. Dilute with about 10 ml. water and filter through *lead-free* paper. Neutralise with AmHO and determine as above.

Beer. Detection. Dilute 10 ml. to 50, add 5 ml. E/10.HA, and 3 ml. H₂S solution. If lead exceeds $\frac{1}{2}$ grain per gallon there will be darkening, or if $\frac{3}{4}$ grain of copper or tin be present.

Determination. Concentrate 100 ml. in a porcelain dish that will go into a muffle, add 10 ml. HNO₃ and continue evaporation till viscous. Well mix 1 gm. of lead-free magnesia, dry and ignite in muffle. Treat ash with 3E.HNO₃, neutralise with AmHO, and determine the lead with H₂S (Knapp, *J.S.C.I.*, 1911, 165).

COPPER

The use of copper to preserve the colour of peas and other green vegetables has been the cause of much debate in the Law Courts. Although a Departmental Committee, in 1901, recommended "That the use of copper salts in the so-called greening of preserved foods be prohibited," no Government Regulations were made till 1925, after the report of another Committee. These Regulations only prohibit the *addition* of copper compounds for colouring, and have no relation to any copper *naturally* present in a food. This fact was overlooked in two prosecutions, in 1927, for dried peas, when the amount of copper present was not inconsistent with it having been present naturally (p. 315).

Vedrödi (*Analyst*, 1896, **21**, 235) found 10–710 parts of copper per million in cereal grains, 60–320 in leguminous seeds, and 230–1,350 in capsicum. Lehmann (*Analyst*, 1896, **21**, 290) considered Vedrödi's results were too high. He found the species of the plant had far less influence than the quantity of copper in the soil. Lindow, Elvehjem and Peterson have determined the copper content of a variety of foods (*S.P.A.*, 1929, **54**, 420).

The Report of the Preservatives, etc., Committee for 1901, contains medical evidence and experiments as to the effects of copper

on the human body. Drummond (*S.P.A.*, 1925, **50**, 481) has reviewed the subject and given experiments on rats. Thresh (*Lancet*, 1925, March 28 ; *Analyst*, 1925, **50**, 248), after consultation of the authorities, is of opinion that copper is vastly less deleterious than lead, and that, after the exclusion of cases in which normal proportions of copper in the body have been wrongly blamed, "there is no evidence of copper in small quantities being a poison or of its being able to produce a condition of chronic poisoning."

In addition to the prosecution for peas, beans and spinach, there have been cases in which the accidental presence, rather than the intentional addition of copper, has given rise to prosecutions for lemonade and ginger beer. The L.G.B. Report for 1911 mentions a case of a sauce contaminated with copper from verdigris on the heating coil.

Bodmer and Moor (*S.P.A.*, 1897, **22**, 141) have discussed four methods for the determination of copper in peas, etc., and given the amounts found. Jamieson (*Analyst*, 1919, **44**, 214) and Trotman and Sutton (*S.P.A.*, 1924, **49**, 271) have given methods for the determination of copper (and lead) in gelatin. Lampitt, Hughes, Bilham and Fuller (*S.P.A.*, 1926, **51**, 327) have studied in detail the ferrocyanide and quinosol methods for determining copper in foodstuffs. Callan and Henderson prefer sodium diethyldithiocarbamate to ferrocyanide for the determination (*S.P.A.*, 1929, **54**, 650). Elvehjem and Lindow (*Analyst*, 1929, **54**, 245) and Richardson (*S.P.A.*, 1930, **55**, 323) have given methods for determining minute quantities. See also Chalk (*S.P.A.*, 1930, **55**, 187). Stevenson (*S.P.A.*, 1897, **22**, 87) has given a warning against the use of brass burners in ashing for copper determinations.

The possibility of copper being mistaken for lead has been alluded to previously. The tinctorial power of copper sulphide is much less than that of lead sulphide ; allowing for this, in some cases in which both metals are present, copper may be determined by comparison of the results obtained in acid solution and in alkaline solution after the addition of KCN.

Copper may be *detected* in peas, beans, etc., by mashing with E.HCl, and putting in a piece of bright steel wire ; 0.5 grain per lb. may show a deposit of copper in an hour, and 0.1 grain will show on standing all night.

It may be *determined* as follows :—Dry 70 gm. of the crushed peas on the water bath in a flat-bottom porcelain dish. Moisten with about 7 ml. H_2SO_4 and heat till frothing ceases. Gradually ignite in muffle and treat ash with 25 ml. 7 v/v HNO_3 , and filter into large platinum dish. Ignite filter paper in porcelain dish, treat with a few drops of HNO_3 and H_2SO_4 , then with about 70 ml. of the 7 v/v HNO_3 and filter into the dish.

Deposit the copper on the dish by an electric current of about

2 volts and 0.2 amperes passing all night. Syphon off the liquid while the current passes, pour in water and syphon out separately. Break the circuit, wash with water and twice with methylated spirit, then hold with tongs and heat gently with a naked flame. When cold weigh the dish, leaving it on the pan till the weight is constant, say two minutes. Dissolve the copper with HNO_3 , wash, dry and weigh the dish as before. The liquid first syphoned off should be tested for copper, and if necessary the trace present determined colorimetrically. The amount of copper in the liquid strained from the peas may be determined similarly, 140 ml. or more being evaporated with about 5 ml. H_2SO_4 .

TIN, CANNED FOODS

In 1880 Hehner examined a number of tinned foods (*S.P.A.*, 1880, **5**, 218) and found tin in most of the animal and vegetable foods tested. A soup had 0.5 grain of tin per lb., and a sample of oysters 0.7 grain tin, and a considerable quantity of copper. In 1883, Wynter Blyth (*Analyst*, 1883, **8**, 161) reported that fruits, apricots, pineapples, and tomatoes preserved in tins contained from $1\frac{1}{2}$ to 11 grains of tin per lb. In 1894, Fairley examined samples of tinned pears which had caused violent sickness (*F. & S.*, 1894, 322). One of them contained $4\frac{1}{2}$ grains of zinc, and $\frac{1}{2}$ grain of tin per lb. The largest amounts of tin detected by Colwell and Parkes (*B.F.J.*, 1901, 146) in 15 samples of tinned fruits were 0.88 grain per lb. in pineapple, and 0.72 grain each in pineapple and pears. The Report of the L.G.B. for 1906 remarked that 16 % of the samples of tinned food examined that year contained boric acid. The Report for 1909 stated that a sample of tinned pineapple contained 8.6 grains of tin per lb., and one of black cherries 1.96 grains of lead per lb.

O. Jones (*S.P.A.*, 1923, **48**, 429) has given a method for the examination of tinplate; he considered that a suitable specification was 0.75 gm. of tin per sq. dcm. He found that zinc chloride used as a flux in soldering has a marked effect on the amount of tin taken up by the food. The meat in an unsoldered can after a year contained 0.0015 % of tin, and that in a soldered can 0.0103 %. In two years an unsoldered can increased the amount of tin present from 0.001 % to 0.0021 %. He found some tin present in every sample; fruits and vegetables had sometimes 0.027 %. Davies has shown that a molybdenum test may be applied to detect tin in dark patches in cheese (*S.P.A.*, 1932, **57**, 95).

Monier-Williams (*S.P.A.*, 1926, **51**, 402) found that about two-thirds of the gas in canned loganberries which were "blown" was hydrogen. In places the internal lacquer was defective, and there had been extensive corrosion with solution of tin and iron.

In 1908 Buchanan and Schryver reported on the presence of tin in canned goods (Report No. 7 of Inspector of Food ; Abstract, *Analyst*, 1909, **34**, 121). The latter took daily doses of a soluble tin salt for three weeks, in increasing quantities. Little of the tin was absorbed and no ill-effects followed. Buchanan remarked that quantities of tin approximating to 2 grains per lb. are not only unusual and unnecessary, but must be regarded with grave suspicion.

The effects of tin on the body depend on the solubility of the tin salt ingested. Hehner (*loc. cit.*) found that while stannic hydrate had little effect on guinea-pigs, stannous hydrate was a powerful irritant poison. Goss (*Analyst*, 1917, **42**, 208) stated that when tin was dissolved, the greater part of it was converted into an insoluble and stable complex, probably in combination with proteins ; and that it did not have the physiological effects of soluble tin salts. O. Jones (*loc. cit.*) found that usually the tin was in an insoluble form, but that in some cases most of it was in solution. A sample of beef five years old examined by Wirthle (*Analyst*, 1900, **25**, 207) contained 0.0325 % of tin, and the liquor 0.014 %. Sir W. Wilcox has recently stated that the danger of metallic poisoning from the eating of tinned food is practically *nil*, and that he had never met a case (*Grocer*, 1929, Dec. 21).

The official reports for England and Wales give figures which show that the percentages of adulteration were as follows. The Preservative Regulations came into force in 1927 :—

	1906-13	1920-6	1927-30
Canned meat . . .	8.1%	6.1 %	1.3 %
Canned fish . . .	5.3 %	5.9 %	5.4 %

DETERMINATION. Hehner (*loc. cit.*) determined tin by heating the ash with HCl, boiling off most of the acid, boiling with water and filtering. The alternate treatment with acid and water was repeated until no further precipitate was obtained with H₂S. Two Birmingham samples of tinned beef gave 0.2 and 0.4 grain of tin per lb. A sample of apricots when freshly opened had 0.8 grain, and after three days in the tin 2.5 grains of tin. Hehner pointed out that lead is not readily dissolved in the presence of tin, and that there was a possibility of brown stannous sulphide being mistaken for lead sulphide. Manicke and Lauth (*Q.J.P.*, 1928, 110) emphasise the importance of destroying all organic matter, as it adsorbs tin with great tenacity.

Schryver (*loc. cit.*) determines tin by heating 25 gm. with 25 gm. K₂SO₄, and 25 ml. H₂SO₄ mixed with 100 to 150 ml. water, in a flask. After gentle heating to avoid frothing, 25 ml. of H₂SO₄ is added and heating continued till decolourised, but not allowed to go dry. The diluted solution was precipitated with H₂S, dissolved in NaHO, reprecipitated with HÄ, and weighed as oxide. Glassmann and

Barsutzkaja have described a volumetric method for its determination in jam, etc. (*Analyst*, 1929, **54**, 110).

PROSECUTION FOR TINNED RASPBERRIES. *Southport.* Dissolved tin 3.04 grains per lb., instead of a maximum limit of 2 grains. The Medical Officer of Health considered that the article might cause acute irritant poisoning, with vomiting and diarrhoea. The tin was said to have been in stock four months, and had probably been packed the previous year. Fine £5 (*Grocer*, 1922, Feb. 18; *B.F.J.*, 1922, 29).

PROSECUTION FOR TINNED TOMATOES. *Southport.* Dissolved tin $5\frac{1}{2}$ grains per lb., anything in excess of 2 grains was considered dangerous. The Public Analyst stated that he usually found $\frac{1}{2}$ to 1 grain per lb. Tomatoes were particularly dangerous, because they seemed to contain more acid than other fruits. The tin from which they were taken was old and badly corroded. The vendor undertook to throw out the remainder of the goods and was only ordered to pay costs (*Grocer*, 1923, Jan. 13; *B.F.J.*, 1923, 3).

PROSECUTION FOR TINNED ASPARAGUS. *Dover.* Tin 2.9 grains per lb., from which the liquor had been drained away. The Medical Officer of Health for another district, who was an expert on food poisoning, stated that a great deal of the tin in tinned foods was insoluble, and that most of it passed through the body without doing harm. He had collected over fifty cases of food poisoning ascribed to canned foods, and not one of them was due to tin. Case dismissed (*Grocer*, 1923, May 19; *Analyst*, 1923, **48**, 326; *B.F.J.*, 1923, 53).

PROSECUTION FOR TINNED SMALL FISH IN TOMATO. *Greenwich.* Tin 0.071 %. The magistrates considered the sale to be an innocent one, and ordered the defendant to pay 42s. costs (*Grocer*, 1923, June 23; *B.F.J.*, 1923, 80).

PROSECUTION FOR TINNED SARDINES. *London, Old Street.* Tin 3.15 grains per lb., being an excess of 1.15 grains. Dismissed, as warranty proved (*B.F.J.*, 1931, 69; *Grocer*, 1931, May 30).

ZINC

Zinc may be present in drinking water, and galvanised iron kettles have been sold for use with a soft water in place of iron kettles. A peculiar silky turbidity is often seen in such waters. Zinc has been found in preserved peas, probably as a substitute for copper, in evaporated apples due to drying on galvanised iron trays, and in vinegar.

A case of mass poisoning by zinc is given in the *British Medical Journal* (1923, 201; *Analyst*, 1923, **48**, 184); 200 out of 400 people who partook of stewed fruit were ill within a few minutes, but soon recovered. The fruit was found to contain 7 grains of zinc oxide

per lb. Rinck (*J.S.C.I.*, 1914, 883) has described physiological tests which showed that 7–8 mgm. of zinc per litre taken over a long period gave no indication of injurious results. Heller and Burke (*Analyst*, 1927, **52**, 554) found that zinc added to the normal ration of rats did not disturb normal functions through three generations.

Zinc may be determined in water as follows:—Add 10 ml. 3*N*.HCl and 1 ml. fresh 1 % solution of $K_4Fe(Cy)_6$ to 50 ml., or more, of the water in a cylinder. Standards should be similarly made from a zinc solution containing 0.001 per 1 ml., not more than 2 ml. being used. Comparisons should be made after five minutes, the cylinders being placed half over a strip of black paper. Iron, if present in more than traces, should be previously removed by precipitation with ammonia, but unless excess of iron is present phosphate of zinc may be precipitated (cp. Meldrum, *Analyst*, 1918, **43**, 72).

For the determination of zinc in foods the organic matter should be destroyed by H_2SO_4 and HNO_3 . Bodansky (*Analyst*, 1921, **46**, 518) has given a general method for foods; Trotman and Sutton (*S.P.A.*, 1924, **49**, 274) one for gelatin, and Cribb and Still (*S.P.A.*, 1925, **50**, 286) one for peas.

ALUM

The usefulness of alum for obtaining a whiter and better-looking loaf appears to have been well known at the beginning of the nineteenth century, as Bread Acts were passed in 1822 and 1836 which limited the constituents which might be used in bread-making. Powers of search for unlawful ingredients were also given. In 1855 Tennyson wrote :

“ . . . chalk and alum and plaster are sold to the poor for bread,
And the spirit of murder works in the very means of life.”

The report of the Local Government Board for 1877 observes that *only* 7.4 % of the samples of bread were adulterated with alum, and 6 % of flour.

While alum in bread appears to have been generally recognised as injurious to health, its presence in baking powder caused keenly contested prosecutions. It was argued that while alum itself was injurious, in the presence of the alkali in baking powder harmless alumina was formed. In 1879 Mott (*Analyst*, 1879, **4**, 143) found many American baking powders were adulterated with alum, and in the next year (*Analyst*, 1880, **5**, 160) he published experiments which showed that alumina in various forms given to dogs produced sickness and constipation and retarded the digestion of fibrin and white of egg. West-Knights (*S.P.A.*, 1880, **5**, 67) also showed that alum baking powder diminished the solubility of gluten and bread, and retarded the action of diastase.

Experiments by Hehner (*S.P.A.*, 1892, 17, 201) indicated that alum baking powder retarded the digestion of white of egg, milk and bread to a similar extent to alum itself, but differed from alum by having hardly any action on the digestion of flour. Hehner and three of his assistants took doses of alum baking powder and suffered from indigestion; discomfort for several days followed.

In some of the early prosecutions the opinion of Liebig was followed—that part of the injurious effects of alum were due to the removal of soluble phosphates in the form of insoluble phosphate of alumina. This was probably due to a misapprehension, as Hamill (Food Report to the L.G.B., No. 12, 1911, p. 32) has pointed out that a large portion of the phosphorus in flour is in organic combination and not as potassium phosphate, which is present in the ash of flour. In a later prosecution eminent medical evidence was given that the precipitated hydrate of aluminium would be redissolved in the body and that the chloride of aluminium formed would produce similar gastric and other disturbances to alum itself (*F. & S.*, 1893, April 15). In 1894 a conviction for selling a baking powder containing 40 % of alum was quashed by the High Court (*James v. Jones*). According to this curious decision baking powder which contained 40 % of ground rice was not a “food” because it was not taken directly as food, although anyone making bread from it was liable to conviction. Adulteration of baking powder with alum accordingly continued unchecked until 1899, when the legal definition of “food” was extended.

DETECTION. In 1878 there was discussion as to the value of the logwood test for alum, and Young published several papers on the subject (*S.P.A.*, 1879, 4, 6; 1887, 12, 29, 145). He showed that genuine sour breads, or breads to which acetic acid had been added, gave the reaction for alum, and that in natural flour alumina is associated with the gluten. Colwell and Parkes (*B.F.J.*, 1900, 346) have shown that logwood is very sensitive to lead or iron, 1 in 10,000 giving a decided blue colour.

The writer applies the test as follows :—Prepare a 2 w/v solution of logwood by boiling, decanting and cooling. Mix about 10 ml. of it with about 10 gm. of flour or bread in a small beaker, then add 1 ml. of 5E.Am₂CO₃ solution, and mix. Genuine flour will give a pink colour, which becomes brown on exposure to the air. One grain of alum, or 70 grains of calcium acid phosphate, per lb., will show a blue colour after a day, after which the blue of alum tends to fade and that of superphosphate to increase. If a thin film be heated, the blue remains if alum alone be present, but it is destroyed in the presence of superphosphate. The logwood solution should be fresh, and should not be mixed with the alkaline solution before use.

As there have been cases in which calcium superphosphate has

been wrongly reported as alum, it may be worth while to point out differences. The AmHO precipitate of calcium phosphate is insoluble in KOH solution and is gelatinous. The precipitate dissolved in a slight excess of HCl , deposits CaSO_4 on addition of H_2SO_4 and two volumes of alcohol. Schoeller and Webb have given a method for the separation of traces of alumina from iron (*S.P.A.*, 1929, 54, 711).

DETERMINATION. As alumina is neutral to litmus, the amount of (SO_3) combined with it in alum can be determined by that indicator (cp. Tingle, *Analyst*, 1921, 46, 343). On the other hand, as alum is neutral to methyl orange, the amount of NaHCO_3 can be determined in its presence.

Bread. The following, which is a modification of the Normandy process, gave a correct result when 151 grains of alum had been added per 4 lb. loaf of bread :—Dry 100 gm., ignite at a moderate temperature, and fuse powdered ash with Na_2CO_3 and a little KNO_3 . Remove and weigh SiO_2 . Add dilute NH_3 to filtrate almost to precipitation, then a slightly acid solution of $\text{AmC}_2\text{H}_3\text{O}_2$ and boil. Filter out the precipitate after standing, dissolve it in the minimum amount of HCl , and add to a large excess of NaHO solution (free from alumina) in a platinum dish. If the iron precipitate is greyish red, it should be reprecipitated. Acidify filtrate and add $\text{AmC}_2\text{H}_3\text{O}_3$, a little Na_2HPO_4 and a slight excess of NH_3 . Heat, filter and ignite AlPO_4 . After calculation to percentage of alum, the percentage of SiO_2 found is subtracted, and multiplication of the difference by 280 gives grains of alum per 4 lb. loaf. The calculation is based on the assumption that the amount of natural alumina in flour is proportional to the silica present.

Baking Powder. In alum baking powders, in which the amount of iron and calcium present is negligible, the amount of P_2O_5 present may be subtracted from the alumina precipitated by ammonia, and the difference calculated to alum. Determination of NH_3 will indicate if ammonia alum has been used, and may give a confirmation of the proportion present. The use of potash alum is less likely.

CHAPTER IX

PRESERVATIVES

Prosecutions. Salicylic acid, detection, prosecutions. Benzoic acid. Sulphur dioxide, detection, determination. Boric acid. Borax powder, prosecutions. Boron preservatives, analyses, detection and determination in foods.

PREVIOUS to 1896 there was no examination for preservatives in Birmingham other than for excess of salt in beer. In that year, at the request of the Public Health Committee, the Public Analyst (Alfred Hill) tested a number of foods for preservatives. As the result of these investigations he publicly advocated prohibition of the use of preservatives in food. At a conference of the Sanitary Institute held in Birmingham in 1898, he took that subject for his presidential address to one of the sections and the next year gave a paper before the Incorporated Society of Medical Officers of Health and a resolution was passed deprecating the use of preservatives in foods. In the same year he gave evidence as representative of that Society before the Departmental Committee which enquired into the use of preservatives and colouring matters. In 1901 that Committee made a report and recommended limitation or prohibition of preservatives in food. Unfortunately no legislation resulted, and although prosecutions for preservatives in milk were instituted under the Sale of Food and Drugs Acts, it was not until 1912 that the Milk and Cream Regulations definitely prohibited the use of preservatives in milk.

In 1923 another Departmental Committee was appointed and that Committee finally reported in 1924. In the following year the Public Health Preservatives, etc., in Food Regulations were passed which prohibited the use of boric acid, formic aldehyde and salicylic acid, and only permitted the presence of sulphur dioxide (sulphites) and benzoic acid in certain cases and in limited quantities, and also prohibited the use of metallic colouring matters and several dyes.

In the following chapters, particularly under milk, butter, cream and cake, particulars are given of the action that has been taken and of the great diminution in the use of preservatives that has taken place. It will be seen, I think, that Birmingham has taken an honourable place in the fight against what has been described as "embalmed foods."

Some makers of food preservative have, in the past, been eloquent in praising their wares. In 1904 a preservative used by some milk

vendors caused them to be prosecuted and fined. The following are quotations from the advertisement : —

“ —’s milk preservative cannot be regarded in the light of an adulterant, since it is pure, wholesome, and quite harmless.”

“ It may be used with every confidence—it has never been known to fail—it does its work thoroughly well without the aid of drastic doses of objectionable chemicals : in a word —’s milk preservative marks a distinct advance in science, and is being used with the greatest success everywhere.”

“ Scientists, analysts and chemists of the highest standard have approved of it, and it is, as has been truly said, ‘ reliable and efficient ; innocuous and tasteless.’ ”

This “ distinct advance in science ” was only boric acid and borax !

The new Preservatives Regulations prohibit any such advertisement and require that articles sold as preservatives shall bear a label stating the percentage of sulphur dioxide or benzoic acid present.

PROSECUTIONS. *Brentford.* A firm of chemical manufacturers was summoned for : (i.) Selling “ Preservative powder, 7 % sulphur dioxide ” which contained an excess. (ii.) Giving false warranty. (iii.) Failing to state on the label the true percentage of sulphur dioxide present. The powder contained 11.1 % of sulphur dioxide. The magistrates considered the first offence was not proved, but inflicted 40 gns. fines on the other two (*P.J.*, and *Grocer*, 1927, Nov. 19 ; *B.F.J.*, 1927, 119).

Barnsley. Selling a “ Milk preserver ” which contained boric acid. The chemist was entitled to sell boric acid but not to recommend its use in milk. The defendant was ordered to pay costs only, the magistrates thinking there had been a misunderstanding (*P.J.*, and *Grocer*, 1928, Dec. 15 ; *Analyst*, 1929, **54**, 106 ; *B.F.J.*, 1929, 6).

Nottingham. Selling “ Iceit ” in terms likely to lead to it being used as a food preservative. It contained 94.1 % of boric acid, and 2.08 % of borax. Fine £5 (*B.F.J.*, 1929, 100).

SALICYLIC ACID

Pure salicylic acid should have a melting point of 158–9° C., a lower figure may be due to the presence of para-cresotic acid, which has a melting point of 151° (Hesse, *P.J.*, 1889, xx. 174 ; Dunston and Bloch, *P.J.*, 1900, xxi., 429 ; Charteris, 1900, 436 ; Hill, 1900, 479 ; Abstracts, *Y.B.P.*, 1891, 28, 31).

Papers on the estimation of salicylic acid have been given by Harvey (*S.P.A.*, 1903, **28**, 2), Harry and Mummery (*S.P.A.*, 1905, **30**, 124), and von Fellenberg (*Analyst*, 1910, **35**, 437). A method for milk and cream has been worked out by Revis and Payne

(*S.P.A.*, 1907, **32**, 286). Dubois considers that carbon disulphide is more satisfactory for extracting it from wines (*Analyst*, 1907, **32**, 220). For alcoholic beverages, see Daels (*Q.J.P.*, 1929, 83). Monier-Williams has applied his process for benzoic acid to salicylic acid (Public Health Report, No. 39, 1927, p. 47). Lerrigo has shown that salicylic acid after extraction with ether is dry in forty-eight hours at air temperature, and in two hours at 35° C. (*S.P.A.*, 1926, **51**, 79).

Nicholls has found that ferric chloride gives the best colour with salicylic acid if the acidity is N/500; he recommends Jorissen's test (*S.P.A.*, 1928, **53**, 20). Sherman (*Analyst*, 1910, **35**, 252) and Backe (*Ibid.*, 253) have given a warning that maltol, in baked bread, etc., gives a reaction simulating salicylic acid.

Henville states that salicylic acid may be determined in sodium salicylate by titration in the presence of ether (*S.P.A.*, 1927, **52**, 149).

There have been prosecutions for the presence of salicylic acid in cordials, ginger wine, ipecacuanha wine, lager beer, raspberry vinegar, lemon squash, mineral water, lemon cheese and curd, jam, jelly, mincemeat, and syrup of figs. Details are given under these headings.

DETECTION, ETC. A standard solution 0.01 % will only keep a day or two, but 0.1 % is fairly stable. A solution 0.057 % is sometimes useful; it contains 5 grains per pint. The salicylate of iron colour, when partially destroyed by HCl, may be removed by ether, both solutions being colourless, but ether will not remove salicylic acid from salicylate of iron if the solution is neutral. Citric or tartaric acid will prevent the formation of the colour. The following tests will detect 0.01 % of salicylic acid, in each case E/5 FeCl_3 is the reagent.

Marmalade. Mix about 10 gm. in a dish with water, nearly neutralise to phenol phthalein with N/NaOH, dilute to about 50 ml. Decant liquid into a tube and add 1 ml. of reagent at a time.

Butter. (i.) Rub on tile with a knife and a little water and reagent. (ii.) Shake in test tube with hot water, and add reagent. The colour is more apparent in the aqueous layer after standing.

Cream. Shake with about 10 volumes of water and filter. Add reagent to milky filtrate.

Sherry. Take 1 ml., add 5 ml. of water and 5 drops of reagent.

Vinegar. Take 10 ml. in test tube, add 3 drops of 3E.HCl and 5 ml. ether. Shake gently, close the tube with thumb, invert and run off the aqueous liquid. Evaporate the ether in the test tube, add a few drops of water and 1 drop of reagent.

Beer (cp. Muter, *S.P.A.*, 1877, **1**, 193). Mix 10 ml. with 10 ml. of water in Nessler cylinder and add 2 ml. of reagent. Compare the colour with beer to which $\frac{1}{2}$ or $\frac{1}{4}$ grain per pint of salicylic acid has

been added. For determination, put 50 ml. of beer, 5 ml. N/10 HCl, and 5 ml. chloroform into a separator. Shake one minute, run the emulsion into a 1 oz. stoppered bottle, add 10 ml. water, 10 drops of the reagent, and shake. Compared with standards made from pure beer in the same way.

PROSECUTIONS. *Glasgow.* Cresotic acid $2\frac{1}{2}$ %. Evidence was given that cresotic acid was an impurity in some artificial salicylic acid, and that it was poisonous. The case was withdrawn (*Analyst*, 1890, **15**, 220; *P.J.*, 1890, Oct. 25).

Glasgow. Cresotic acid 8 %, supplied in twelve powders. Fine 3 gns. (*F. & S.*, 1893, Feb. 4).

BENZOIC ACID

In Report No. 39 on Public Health and Medical Subjects (1927) Monier-Williams has recorded a number of experiments on the detection and determination of benzoic acid, has summarised published methods, and given a process for its determination. Later on he published another process (*S.P.A.*, 1927, **52**, 572).

Other recent references are: Waltzinger (egg-yolk, *Analyst*, 1927, **52**, 90); Nicholls (determination, *S.P.A.*, 1928, **53**, 19), Daels (alcoholic beverages, *Q.J.P.*, 1929, 83), Harral (detection and determination, *S.P.A.*, 1930, **55**, 445), and Leather (detection, *S.P.A.*, 1931, **56**, 299). (*See Addenda*, p. 577.)

Lerrigo found that there was very little volatilisation of benzoic acid at 40° C. (*S.P.A.*, 1926, **51**, 405). Henville has given a method for the titration of benzoic acid in sodium benzoate in the presence of ether (*S.P.A.*, 1927, **52**, 149), and Chapman has given a warning that caramel may yield an acid which closely simulates benzoic acid (*S.P.A.*, 1927, **52**, 215).

Prosecutions for benzoic acid in the following foods are given subsequently: milk, damson jam, sausage.

SULPHUR DIOXIDE

Soon after the passing of the Preservative Regulations Monier-Williams made a Report to the Ministry of Health on "The determination of sulphur dioxide in Foods" (Report No. 43 on Public Health and Medical Subjects, 1927; Abstract, *Analyst*, 1927, **52**, 415). In this Report he discusses published methods in detail, and proposes one for the purpose which is given below. Subsequently H. O. Jones (*S.P.A.*, 1928, **53**, 138) advocated distillation in a vacuum, and Black and Warren (*S.P.A.*, 1928, **53**, 130) showed that the interference by glucose and gelatin was negligible, but that of mustard, nutmeg and ginger is considerable. A joint report of chemists engaged in food manufacturing industries

(*S.P.A.*, 1928, **53**, 118) should also be consulted. Jensen (*S.P.A.*, 1928, **53**, 133) gave a method without distillation based on Ripper's principle for glucose syrup, sucrose and cornflour.

The determination of sulphur dioxide in beer has been discussed by Baker and Day (*S.P.A.*, 1912, **37**, 439), in gelatin by Trotman and Sutton (*S.P.A.*, 1924, **49**, 271), in dried fruits by May (*S.P.A.*, 1927, **52**, 271) and Miller (*S.P.A.*, 1927, **52**, 338), and in fatty substances by Knapp and Phillips (*S.P.A.*, 1928, **53**, 149). Henville has described a new apparatus for the detection and determination of sulphur dioxide; he considers loss is due to volatilisation, rather than oxidation (*S.P.A.*, 1929, **54**, 228). Herd has obtained greater accuracy by using barium benzene sulphonate instead of barium chloride (*S.P.A.*, 1930, **55**, 35).

Particulars of prosecutions for sulphur dioxide in meat, sausage, ginger, dried fruit and peel, pearl barley, confectionery, beer, and malt vinegar are given subsequently under the various foods.

DETECTION. After Parkes (*S.P.A.*, 1921, **46**, 402; 1926, **51**, 620). Put 10–20 gm. of the material into a 200–300 ml. flask with a lump of marble and about 20 ml. 3E.HCl. Close flask with a safety funnel having two bulbs, which contain 2–3 ml. of a solution which has been prepared by adding to E/50 iodine solution about one-fifth of its volume of E.BaCl₂, and which has been filtered after standing. After heating on the water bath for about fifteen minutes in the presence of SO₂ the iodine will be more or less decolourised, and BaSO₄ precipitated.

DETERMINATION. After Monier-Williams (*loc. cit.*). The apparatus used is a litre flask closed with a bung through which passes: (1) a tube connected with a Kipp apparatus generating CO₂ from marble and pure HCl, and (2) a tube with a bulb attached to a slanting condenser, the upper end of which is connected with a flask, the cork of which also carries a tube connected with a three-bulb U tube.

10 ml. of 20-volume hydrogen peroxide solution are mixed with about 15 ml. of water, and neutralised to bromo-phenol blue. This liquid is divided between the U tube and the flask, so that the liquid covers the lower end of the vertical tube.

About 500 ml. of hot tap-water and 20 ml. 10E.HCl are put in the flask and the liquid raised to boiling for a few minutes to remove oxygen, while CO₂ is passed. The flask is cooled, and to it are added 100 ml. of beer, or 50 gm. of sausage or other solid. Distillation is continued for about an hour, during which CO₂ is passed. At the end of that time the water supply to the condenser is turned off, and the condenser allowed to become hot. The liquids in the flask and the U tube are mixed with the washings of the vertical tube and titrated with N/10.NaHO, using bromo-phenol blue. 1 ml. = 32 parts per million of SO₂, if 100 ml. have been taken.

BORIC ACID

The titration of each of the 140 Birmingham samples examined, 1910-28, using glycerin, gave figures approximating to 100 % of boric acid, the difference from it being almost within the limit of experimental error. The average loss of five samples dried in the water oven was 33.0 %. If H_3BO_3 lost H_2O only, the theoretical loss would be 29.1 %. The loss on drying appears to be an indefinite figure, boric acid being volatilised in addition to the water lost.

The B.P. limit for arsenic is 5 parts per million. Of the 127 samples tested 1918-31, 96 % contained 0.2 parts, the other 4 % 3-5 parts of arsenic per million.

SOLUBLE LEAD IN BORIC ACID (165 samples)

Parts lead per million	0-5	-10	-15	-20	-25	26-100	Total
1907-23.	14	23	23	20	6	14	100
1924-31.	40	27	13	11	6	3	100

The improvement in the second period is notable, as only 3 % of the samples exceeded the B.P. limit (25 parts per million) against 14 % in the first period : 93 samples, 1920-9, were tested for insoluble lead, 86 had 0.2 parts per million, and 7 had 3-6 parts.

Of the samples examined in England and Wales, 1920-30, 2.9 % were condemned as adulterated.

There appear to have been no prosecutions for adulterated boric acid.

BORAX POWDER

Adulteration of this article is unusual, and more probably due to accident than intention. In 1898 three connected Birmingham samples contained 25-36 % of sodium bicarbonate. Many samples, however, have been condemned because of impurities.

Analytically, borax is half neutralised boric acid, and can be titrated either with acid, using methyl orange, or with alkali, using phenol phthalein and glycerin. Usually the differences between the two titrations is under 1 % of borax. One of the above-mentioned samples gave 142 % and 62 % of borax, the reason being that 36 % of sodium bicarbonate was present, which increased the acid titration but not the alkali one, as NaHCO_3 is neutral to phenol phthalein. Should sodium carbonate be present, the alkali titration may be much lower than the acid, as its alkalinity will affect the two titrations in opposite directions. When carbonate is present the better plan is to boil with excess of acid, neutralise to methyl orange, and then titrate with alkali, etc. (cp. p. 102, and Liversseege, B.P. Conf., 1906, 265).

Calcium borate comes from volcanic regions, and naturally contains arsenic. Borax prepared from it requires repeated

crystallisations to remove this impurity. In the B.P. of 1914, the medicinal article was first described as "Purified borax," and a limit of 5 parts of arsenic per million was prescribed. Previous editions had used the term "Borax," and required that it should yield no characteristic reaction with arsenic.

ARSENIC IN BORAX (41 samples)

Parts arsenic per million	0-1	2-5	7-20	35-100	130,200	Total
1910-26	34	12	17	22	15	100

A number of the samples were commercial borax and quite unsuitable for internal use, though the labels "Refined" and "Finest English" were used on some of them. Borax for commercial purposes is neither a food nor drug, and it is reasonable to expect that it should, particularly when sold in small quantities, bear a label warning the purchaser that it should not be used medicinally.

The B.P. of 1914 required "Purified borax" not to exceed 5 parts of lead per million; 31 Birmingham samples complied with this in 1910-26, and 3 contained 6-8 parts of lead.

Borax first appeared in the official returns for England and Wales in 1901, when 45 % of the samples were condemned. During 1905-13, the proportion fell to 12.3 %; during 1920-4, it rose to 20.0 %, but fell to 3.1 % during 1925-30.

PROSECUTIONS. *Birmingham.* Sodium bicarbonate 35 %, 35 %, and 25 %, respectively. The three defendants had each been supplied with halfpenny packets by one wholesale dealer, and were fined 5s. (*F. & S.*, 1898, Aug. 13).

Wednesbury. Bicarbonate of soda 100 %. The defendant said he thought bicarbonate of soda had been asked for, and was fined £4 for what the stipendiary described as reckless trading (*B.F.J.*, 1899, 118).

Paisley. Arsenious acid about $\frac{1}{2}$ a grain per lb. At an adjourned hearing the authority withdrew the prosecutions, as an error had been made in the analyses. Each defendant was allowed 5 gns. costs (*B.F.J.*, 1901, 91).

Bedford. Carbonate of soda, or ordinary washing soda 50 %. For the defence it was pleaded that the soda had been added by mistake. Fine 2s. 6d. and 10s. costs (*P.J.*, 1912, June 29).

Birmingham. Arsenic 200 parts per million. One sample was bought from a drysalter and was labelled "Borax." The magistrates ruled that the witness asked for "Borax" and obtained that substance, and dismissed the prosecution. A second sample had been sold by a chemist who was a customer of the drysalter, and who, considering it to be pure, had labelled it "Finest English Borax." In this case a fine of 5s. was inflicted (*P.J.*, 1920, Dec. 18).

Durham. Arsenic, not less than 10 parts per million. It was sold by a grocer, and labelled "Finest borax for all household purposes." Fine 10s. (*P.J.*, 1920, Dec. 18).

London. Arsenic 120 parts per million. It had been sold as "Purified borax" at a drug stores. It was pleaded for the defence that except for one case in a thousand, the commercial borax supplied was suitable for ordinary uses—hair washes, toilet preparations or laundry work; and that in the remaining case it was inconceivable that the small amount of arsenic could do harm. The magistrate was told that the borax had been destroyed, and fined the defendant £2 (*P.J.*, 1924, Sept. 27).

Huddersfield. Arsenic 70 parts per million. Sold as "Purified borax." The Medical Officer of Health considered that its continual internal use would be harmful. The chemist explained that commercial borax had accidentally been put in the purified borax container. Fine £5 (*P.J.*, 1928, Dec. 8; *B.F.J.*, 1928, 115).

Wolverhampton. Arsenic 400 parts per million. Sold by a shopkeeper as "Refined borax" in penny packets. Fine £3 (*P.J.*, 1929, June 22).

Leicester. Arsenic 60 parts per million. It was labelled "Finest refined borax," and for the defence it was argued that purified borax was the medicinal preparation, and refined borax a commercial article used for domestic purposes. It was also labelled "Not to be taken internally," and the case was dismissed (*Grocer*, 1931, Jan. 17; *Analyst*, 1931, 56, 181).

BORON PRESERVATIVES

Although the *addition* of boron preservatives is not permitted by the Preservative Regulations, their presence is permissible if they be due to natural causes. Boron is widespread in nature, and its presence has been reported in various foods. It has been found in various fruit juices by Hebebrand (*Analyst*, 1903, 28, 37), in apples and cider by Allen and Tankard (*S.P.A.*, 1904, 29, 301), in Irish moss and agar-agar by Chapman and Linden (*S.P.A.*, 1926, 51, 564), in coffee by Partridge (*S.P.A.*, 1927, 52, 401), and also by Scott Dodd, who found it in cocoa, chocolate, currants, raisins and other fresh and dried fruit, as well as spices and sauces (*S.P.A.*, 1927, 52, 459; 1929, 54, 15). The amounts found have been small, and in some cases open to question, as the determination of traces is a difficult analysis.

ANALYSIS. Monier-Williams has given a paper on the estimation of boric acid in liquid eggs; he preferred magnesia mixture for precipitating phosphates (*S.P.A.*, 1923, 48, 413). The Government Laboratory method for milk, cream, etc., is given (*Analyst*, 1923, 48, 416). The detection and determination of boric

acid has been studied in detail by Scott Dodd in a series of papers (*S.P.A.*, 1927, **52**, 463 ; 1929, **54**, 19, 282, 645, 715 ; 1930, **55**, 23).

Boron preservatives have been made by heating mixtures of boric acid and borax, sometimes in equal quantities, when the percentage of B_2O_3 present is very similar to that in boric acid itself. In such mixtures the alkali titration will be higher than the acid one (cp. p. 99). Ignition with sodium phosphate will give a useful check on the titration, the gain in weight being due to $Na_2B_4O_7$ and B_2O_3 .

The calculations may be made as follows, where M = acid titration, P = alkali one, each without removal of CO_2 , L = acid titration after boiling, Q = subsequent alkali one ; all results being expressed as N.v/w. A = % H_3BO_3 , X = % $Na_2B_4O_7$, 10 H_2O , B = % $NaHCO_3$, C = % Na_2CO_3 , 10 H_2O .

$Na_2B_4O_7$, 10 H_2O %	(without B or C)	= 0.1907 M
„	„ (with B or C)	= 0.0953 Q
„	„ (with C)	= 0.0636 (2P + M)
„	„ (without A or C)	= 0.1907 P
H_3BO_3 %	(with X)	= 0.0618 (P - M)
$NaHCO_3$ %	.	= 0.084 (M - P)
„	.	= 0.042 (2L - Q)
Na_2CO_3 , 10 H_2O	.	= 0.0954 (M - P)
„	„	= 0.0715 (2L - Q)
Increase in weight	.	= 0.5277 X + 0.563 A.

DETECTION. (i.) Put 2 drops of milk, beer or vinegar, or a small piece of butter, cream, cake, etc., in the depression in a white tile. Add 1 or 2 drops of tincture of turmeric, and 1 drop of 3 w/v oxalic acid in E/2.HCl. Heat on top of water oven ; boric acid will give a red colour or tinge. With milk wetting may improve the colour, and approximate quantitative results may be obtained by comparison with standard samples. The turmeric tincture is 10 w/v in industrial methylated spirit, filtered after a day's maceration.

(ii.) Dissolve the ash in a little water, and slight excess of HCl, filter into small flat-bottom porcelain dish, add tincture of turmeric, evaporate nearly to dryness and moisten with the above acid mixture, heat again and observe colour, particularly at the edges.

It may not be superfluous to observe that before any quantitative calculation is made, a positive qualitative test should be obtained.

Determination in Milk. Evaporate 70 ml. of milk in a platinum dish on a water bath with 7 ml. of 3 N sodium hydroxide solution for about four hours. Apply a flame to the side of the dish, taking care that the contents do not froth over. Complete the ignition at a high temperature until the contents are white, or nearly so. Add 35 ml. of water and filter after standing. Add 5 ml. of 3 N

hydrochloric acid to the dish and 15 ml. of water. Filter through the same filter, and wash dish and filter with 4 ml. of 3 N hydrochloric acid mixed with 16 ml. of water. Boil for five minutes, cool, and add 6 ml. 0.5 N calcium chloride solution, phenol phthalein and N sodium hydroxide solution to make the liquid a permanent faint pink. Add another ml. of calcium chloride solution and more sodium hydroxide if the colour is destroyed. Repeat this addition, if necessary (cp. p. 73). Dilute to 100 ml. and filter through a dry filter. To 80 ml. of filtrate add methyl orange and N/10 hydrochloric acid to slight excess. Boil for five minutes, cool, neutralise to methyl orange and titrate with N/10 sodium hydroxide solution and 30 ml. of glycerin. Subtract the acidity of 30 ml. of glycerin.

W/v boric acid = ml. N/10 sodium hydroxide solution $\times 0.011$.

Grains per gallon = ml. N/10 „ „ „ $\times 7.75$.

If more than 5 ml. of sodium hydroxide solution are used, calcium borate may be precipitated, and the precipitate should be re-dissolved and re-precipitated as before (Liverseege and Bagnall, *S.P.A.*, 1924, 49, 133).

Determination in Butter. Weigh 12.4 gm. in small porcelain dish, melt, and pour into small separator. Wash dish into separator with about 50 ml. of boiling water. Shake and stand in warm place till separation occurs. Run out aqueous liquid into 100 ml. flask and wash residual fat with 4 quantities of boiling water as before to make volume up to the mark.

Boil 50 ml. of the liquid, after addition of methyl red, with slight excess of N/10 HCl. Neutralise with N/10 NaOH, add phenol phthalein and 15 ml. of neutral glycerin, and again titrate. Division of the number of ml. used in the second titration by 10 will give percentage of boric acid.

If the end points are not sharp, make the remaining 50 ml. alkaline with NaOH, evaporate to dryness and ignite. Dissolve the residue in water, acidify, etc., as before.

Determination in Cream. If an appreciable quantity of boric acid (0.3 % or more) be present, it may be determined as in butter, but with small quantities the phosphates should be removed as with milk.

Determination in Cake. Wet 12.4 gm. (or more if the proportion of boric acid is small) with 24 ml. E/2.NaOH, evaporate, ignite, and proceed similarly to milk.

Determination in Sausage, etc. Weigh 7.75 gm. of sausage, or 2 gm. of liquid egg, add 50 ml. 1.5E. alcoholic NaOH, cover dish, and put on top of water oven for a few hours, then evaporate carefully, ignite till white, and proceed similarly to milk.

Determination in Boric Acid Ointment. (i.) Weigh 3.1 gm. into a wide-mouth flask, add 100 ml. water, boil and cool. Add methyl

red and neutralise, if necessary. Add phenol phthalein, 25 ml. glycerin, and titrate with $N/2$ NaOH. Each ml. of alkali used equals 1 % of boric acid. (ii.) Weigh 3.1 gm. and treat as butter.

PROSECUTIONS. There have been many prosecutions for boron preservatives in foods, particularly in milk, cream, butter, as well as sausage, fish paste and other nitrogenous food. Particulars are given under the various headings.

CHAPTER X

METHODS OF CALCULATION

Slide-rule. Significant figures. Errors of weighing. Mixtures of two substances, alligation. Dilution. Scales. Graphs. Correction of volumes of gases. Conversion of British and metric ratios. Notation, abbreviations. Sufficiency of a series. Calculation of average from frequency-distribution. Comparative adulteration figure. Standard sampling.

SLIDE-RULE

THE calculation of a gravimetric determination of a nearly pure substance, or when the difference of two products is required, is better done by logarithms, four figures being usually sufficient. For most of the calculations required in food and drug analysis, however, sufficient accuracy (about 1 in 1,000) is attained by the use of a 10-inch slide-rule, which is particularly useful for a series of titrations. Examples of its use may be found in an address by the writer on "The Use of the Slide-rule in Pharmacy" (*P.J.*, 1903, 162).

In many cases the combination of a simple arithmetical process with a slide-rule calculation easily gives greatly increased accuracy. The calculation of milk solids may be given as an example. Suppose 4.987 gm. of milk yield 0.631 gm. of solids. If the weight had been *exactly* 5.0 gm., the milk solids would have been 12.62 %. Actually that percentage requires to be increased in the proportion 13/4987, which is shown by the rule to be 3/1262, which being added, gives the exact percentage 12.65 (*i.e.*, $12.62 + 0.03$).

Similarly, the awkward division resulting from a sp. gr. bottle being slightly under its nominal capacity, may be avoided by adding a fractional part. A sp. gr. bottle held 999.61 grains of water, or 830.73 of spirit. Had the water contents been exactly 1,000 grains, the sp. gr. of the spirit would have been 0.83073. This figure, however, must be increased in the proportion 39/99961, which is shown by the slide-rule to be 33/83073, which being added, gives the correct sp. gr., 0.83106. Five significant figures, though arithmetically correct, are probably not justified by the experiment.

Clark (*S.P.A.*, 1923, 48, 61, 164, 211) has described sliding scales for use when titrating strong solutions by dilution and use of aliquot parts.

THE NUMBER OF SIGNIFICANT FIGURES

The writer has called attention (*S.P.A.*, 1897, 22, 87) to absurdities of statements in published analytical work due to failure

to recognise how far figures introduced by calculation have a real meaning. A more recent example is a certificate for lime-water, which gave "Lime 0.0952 %, Water 99.9048 %," which suggests an accuracy of 1 in a million! A 1929 certificate stated that a ginger contained "1,564 parts of sulphur dioxide per million," and a 1930 one gave 13.881 grains of boric acid per lb.! "The strength of a chain is that of its weakest link" has applications in analytical statement as well as in engineering.

Suppose 10.15 ml. of standard solution be used in a titration and that the indicator is only sensitive to 0.05 ml. In such a case the accuracy does not exceed 5 in 1,000, or 0.2 %. If the substance be nearly pure, the first decimal place is not quite reliable, and a second one meaningless. It also follows that if 10 gm. of the substance be taken for the titration, weighing correctly to 0.001 gm. is an unnecessary waste of time. If the weight be correct to 0.01 gm. the error of titration will not be increased by the error of weighing.

Similarly, the product of the multiplication of two numbers, one of which is correct to 1 in 100, and the other 1 in 1,000, is only correct to 1 in 100, however many decimal places may be produced. This relates to significant figures, whether preceded or followed by ciphers, being independent of the position of the decimal point.

An International Society has suggested that in water analysis the quantities be given to one decimal place only, or, when the quantity is less than 10, to two significant figures (*Analyst*, 1931, 56, 746).

ERRORS OF WEIGHING

It is not always realised that much greater accuracy in weighing is necessary in one part of a determination than in another part.

In a determination of the percentage of ash, or water, in a substance, let " x " be the error in weighing the dish, " y " be the error in weighing the dish and substance, and " z " be the error made in weighing the ash or dried residue, " s " gm. of the substance being taken.

The error in the percentage of ash = $\frac{100(z-x)}{s}$, if $x = 0.01$ gm.,

$z = 0$, and $s = 5$ gm., the error will be 0.20 %.

If an additional error of 0.01 gm. be made in y , the total error will be altered by only 0.002 %.

The error in the percentage of water = $\frac{100(y-z)}{s}$, if $y = 0.01$ gm.,

$z = 0$, and $s = 5$ gm., the error will be 0.199 %, while a similar error in x will hardly affect the result.

Exact weighing of the dish is therefore much more important in the determination of ash than of water. On the other hand, the

substance must be more accurately weighed when water is being determined than for an ash determination.

MIXTURES OF TWO SUBSTANCES, ALLIGATION

The important case of two substances having a common determined constituent may be illustrated by a diagram :—

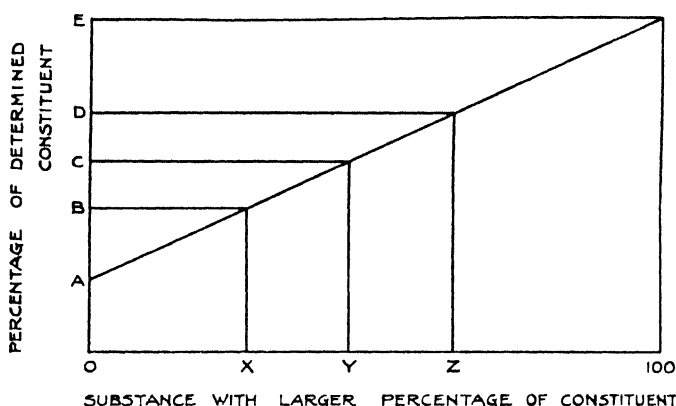


FIG. 1.—Alligation.

The simpler form is when the percentage of the common constituent present in each of the pure substances is known, when the following equation expresses the relation :—

$$\left. \begin{array}{l} \text{Percentage of substance with larger} \\ \text{proportion of constituent.} \end{array} \right\} = ox = \frac{100 (ob - oa)}{oe - oa}$$

To take a concrete example. Dry coffee in certain conditions yields 25 % of solid extract to water, and dry chicory in the same conditions 76 %, while a particular mixture of the two yields 40 % of solid extract. Substituting these values :—

$$\text{Chicory} = \frac{100 (40 - 25)}{76 - 25} = 1.96 (40 - 25) = 29 \%$$

The same result may be obtained with a different arrangement (cp. Fairbourn, *S.P.A.*, 1923, **48**, 263). The three percentages are

put in order with their differences below :—

76	40	25
36	15	—

According to this, there are 15 parts of chicory with 36 parts of coffee, or 29 % as before. It should be noted that the chicory value which is at the *left* above the line is to the *right* below it, and coffee *vice versa*.

In each case it is assumed that no third substance is present, and that the relations between the percentages of the constituent and of the substances lie on a straight line.

In other cases the relationship between the constituent and the mixtures containing it may be known only at points somewhat above and below the proportion in the unknown mixture. In this case the equation takes the form :—

$$\left. \begin{array}{l} \text{Percentage of substance with} \\ \text{larger proportion of} \\ \text{constituent.} \end{array} \right\} = oy = ox + \frac{(oc - ob)(oz - ox)}{od - ob}$$

An example may be taken from a microscopic determination of the proportion of oats and barley. A mixture containing 5 % of barley had a L.E. (lycopodium equivalent) of 12, and one containing 50 % had L.E. 70, while an unknown mixture had L.E. 63. Substituting these values in the equation :—

$$\text{Barley} = 5 + \frac{(63 - 12)(50 - 5)}{70 - 12} = 45 \%$$

As before, no third substance may be present. If the points x , y and z are not far apart, approximate results may be obtained if the relationships are on a slight curve and not on a straight line.

If compositions are expressed as ratios between two constituents, it is necessary to calculate them into percentages before these rules can be applied.

DILUTION

The use of N or E values is convenient for calculating the dilution of solutions. For instance, 3·1E.HCl may be made from 10·1E.HCl by diluting 31 ml. of the strong to 101 ml. If the strength of a spirit solution is expressed as w/v, dilution done in this way will avoid any error due to contraction ; the final temperature should be the same as that of the original alcohol.

If a solution of an intermediate strength (52·1) is to be made by mixing a stronger (62·5) and a weaker (37·9), the values should

be put in order, and the differences below :—

62·5	52·1	37·9
	10·4	14·2

If the strengths are by weight, a mixture of 14·2 gm. of the stronger with 10·4 gm. of the weaker will be the required strength. If they are volume percentages it is better to dilute 14·2 ml. of the stronger to 24·6 ml. (*i.e.*, 10·4 + 14·2) with the weaker.

The same method may be used if the strengths are expressed in terms of sp. gr., though the results will not be exact if any contraction or expansion occurs on mixing.

GRAPHS

The following is an example of a graph for a calculation in which there may be both plus and minus readings, and in which the result

may be expressed in two different ways. Readings in graphs are facilitated by the use of a celluloid set square.

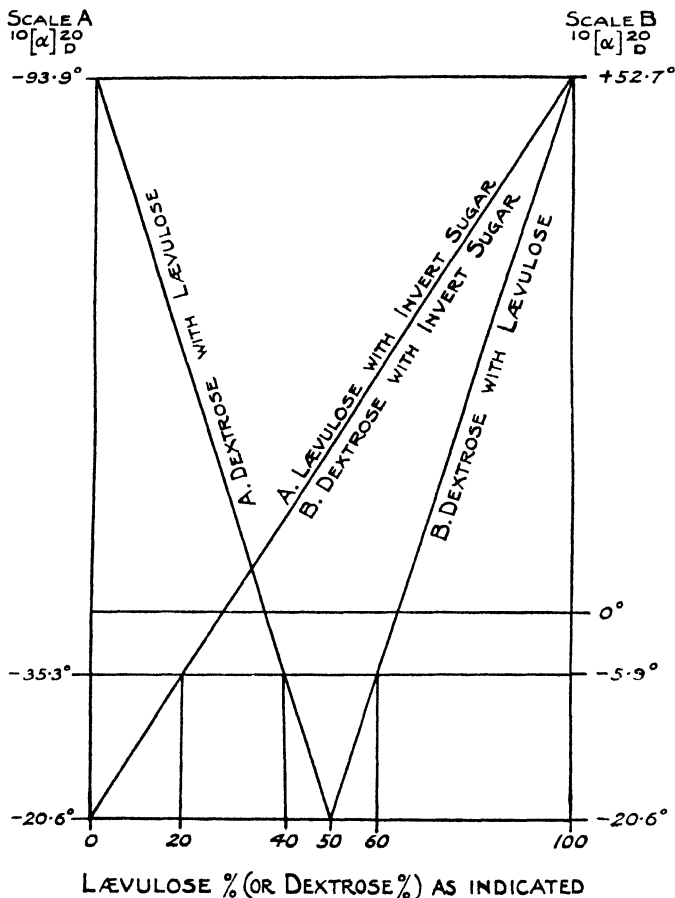


FIG. 2.—Calculation of Sugars (A).

The left scale (A) falls from -93.9° (lævulose) to -20.6° (invert sugar), and the right scale (B) rises from -20.6° through 0° to $+52.7^\circ$ (dextrose). Plotting in this way gives greater accuracy with reasonable size.

$^{10}[\alpha]_D^{20} - 35.3^\circ$ can be read on such a scale as 20 % of lævulose with invert sugar (80 %), or as 40 % of dextrose with lævulose (60 %). Had the specific rotation been -5.9° on the B scale, that reading would have indicated 20 % of dextrose with invert sugar, or 60 % of dextrose with lævulose.

In practice the left-hand lower rectangle has been most used,

and has been plotted on a larger scale, as shown below, which also illustrates the extension of the readings by the use of a third scale :—

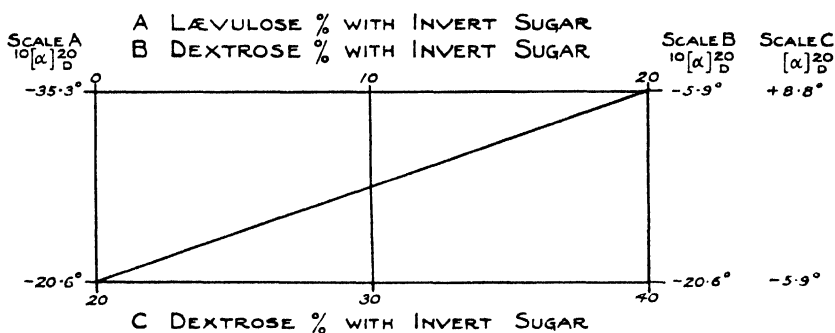


FIG. 3.—Calculation of Sugars (B).

Scale A falls from -35.3° , corresponding to 20 % of laevulose with invert sugar, to -20.6° which is invert sugar. Scale B rises from invert sugar to -5.9° , which is invert sugar with 20 % of dextrose. Scale C rises from -5.9° to $+8.8^\circ$, which corresponds to 40 % of dextrose, in which case the scale at the bottom (20 % to 40 %) is used. This duplication gives greater accuracy with a reasonable size of graph. The paper actually used measures 10×6 inches, each division being $\frac{1}{10}$ inch.

CORRECTION OF VOLUMES OF GASES

In two cases the 1914 B.P. requires liquids to yield definite volumes of gases "at 15.5° and normal pressure." Corrections to these figures are readily made by adding or subtracting definite fractions of the volumes, a selection of which are given below. "Normal pressure" is not defined, but presumably is 760 mm., or corrected for the tension of aqueous vapour 747 mm.

CORRECTION TO 15.5° C. TENSION OF AQUEOUS VAPOUR

Temperature, $^\circ$ C.	8.5	9.8	10.8	11.4	11.9	12.6	14.1
Add 1 volume for each	40	50	60	70	80	100	200
Tension of aqueous vapour, mm.	8	9	10	10	10	11	12
Temperature, $^\circ$ C.	16.9	18.4	19.2	19.7	20.6	21.6	
Subtract 1 volume for each	200	100	80	70	60	50	
Tension of aqueous vapour, mm.	14	16	17	17	18	19	

CORRECTION TO 747 MM. DRY, EQUAL TO 760 MM. MOIST

Pressure, mm., dry	737	735	732	729	726	722
Subtract 1 volume for each	70	60	50	40	35	30

The following is an example of the application of the tables :—

30 c.c. of gas measured at 21.6° C. and 741 mm. pressure.

Correction for tension of aqueous vapour . 741 — 19 = 722

Correction for temperature . . . 30.0 — 30.0/50 = 29.4

Correction for pressure 29.4 — 29.4/30 = 28.4

Coste (*S.P.A.*, 1929, **54**, 656) has described a nomogram for making the correction.

CONVERSION OF METRIC AND BRITISH RATIOS

When it is required to convert a ratio, expressed in British terms, to one in metric ones, much time may be wasted. For instance, gm. per 100 ml. (*i.e.*, w/v), might be expressed as grains per fl. oz. by calculating gm. to grains, then 100 ml. to fl. oz., and finally dividing out.

As a fl. oz. has the volume of 437.5 grains of water, grains per fl. oz. are merely parts by weight per 437.5 volumes, and multiplying w/v by 4.375 will give grains per fl. oz. Similarly grains per gallon are parts by weight per 70,000 volumes.

There will be an error of about 0.2 % in these calculations, as the British standard temperature is 62° F. and the metric 4° C.

SCALES

For calculations which are frequently used it may be worth while to make a scale for working the calculation ; in some cases it may be a permanent copy of a setting of the slide-rule. A scale may be easier both to construct and use than a table. When small values are being converted, particularly if one of them is expressed in vulgar fractions (*e.g.*, parts per million to $\frac{1}{50}$, etc., grain per gallon), such a scale may prevent the error easily made in the position of the decimal point. The divisions on squared paper should be used for one value, and suitable corresponding values of the other, obtained by calculation or from a table, should be inserted ; other values can be interpolated as far as is necessary.

The following (Fig. 4, p. 112) illustrates the conversion of the complicated relationship of butyro-refractometer scale readings to refractive indices, and also a temperature correction (cp. p. 239).

Scales A and C use the lines on the squared paper, and as 15° C. alters the R.I. by 0.0054, the C scale is raised that amount as compared with the A scale. The corresponding B-R. values are inserted from a table, such as that in Evers and Elsdon's " *Analysis of Drugs and Chemicals* " (p. 340). From one value, the

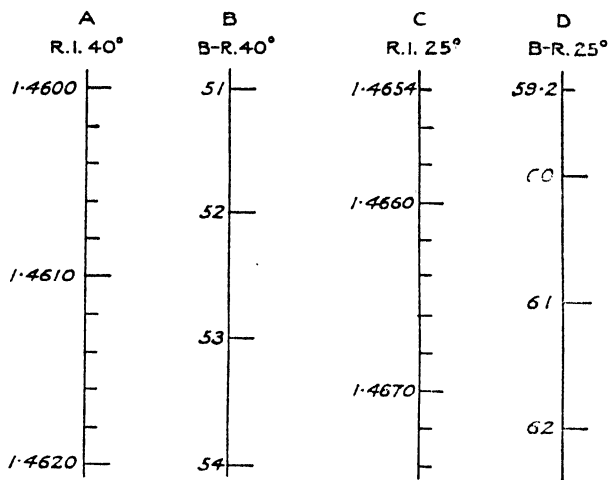


FIG. 4.—Conversion of Refraction Values.

corresponding ones of the three others can be read on the same line.

NOTATION. ABBREVIATIONS

Nearly forty years ago the writer, to avoid the ambiguity that was frequently present in the term “per cent.,” devised the symbols “w/v” and “v/v,” and sent a letter to the *Pharmaceutical Journal* (1895, Feb. 23; see also *S.P.A.*, 1897, **22**, 89) suggesting their use in the following words: “I use the following sign, w/v (weight on volume) for that purpose, reserving % for true percentages by weight, and using v/v for volumes per 100 volumes.” These symbols are now widely used, but often with, as appears to the writer, the superfluous prefix “per cent.” The forms of the symbols suggest percentages without the use of the words. Recently the Pharmacopœia Commission has used the symbol w/w, which is useful for such things as chloroform and alcohol, to indicate definitely percentages by weight.

The writer has also suggested that the symbol v/w may be used with normal, etc., solutions to indicate the volume of an acid or alkali required to neutralise 100 gm. of a substance, or the ash of it. For instance, alkalinity of ash 2.1 N.v/w, or, acidity (methyl red) 2.0 N/10.v/w; similarly for liquids, alkalinity 0.5 N/100, v/v (see *S.P.A.*, 1919, **44**, 28).

THE SUFFICIENCY OF A SERIES

In the determination of such constants as the angle of polarisation or the microscopical lycopodium equivalent, a test is advisable to ascertain if the number of observations is sufficient to give a reasonably constant result. There are two simple tests. (1)

Successive means. The sum of the first two terms is divided by 2, that of the first three terms by 3, and so on. The agreement of the successive means shows if the number of observations is sufficient. In a long series, the last two, the last three, etc., may be treated similarly. Comparison of these with the earlier means will show if the accuracy of the readings is improving.

(2) Divide the series into groups of three or more terms, and compare the means of such groups with each other, and with those of larger groups.

The reliability of a constant may be judged by determining the average of the values found, finding the difference between each value and the mean. The average of these differences, neglecting the sign, is then compared with the constant. When the difference between two values is required, such as the angles before and after inversion of a sugar solution, the percentage accuracy of the difference is much lower than that of the two numbers.

CALCULATION OF AVERAGE FROM FREQUENCY-DISTRIBUTION

The method is best shown by a simple example :—

THE ASH OF GINGER INSOLUBLE IN HYDROCHLORIC ACID

Insoluble ash, %	0-	0.2-	0.4-	0.6-	0.8-	1.0-1.2	Total
No. of samples (frequencies)	7	24	29	18	7	6	91

It is assumed that in each class (0-, etc.) the samples are evenly distributed so that the mid-values (0.1, etc.) fairly represent the average of each class. A value (A) is then arbitrarily chosen about the middle of the table (0.5 in this instance) and the deviations from it, *expressed as multiples of the class intervals* (0.2), are put down in the third column. The products of the frequencies (f) and the deviations (d) are then put down, and the negative and positive products separately added :—

Mid-values of the Class Intervals.	Frequencies. (f)	Deviations. (d)	Product. (fd.)
0.1 %	7	2	— 14
0.3 %	24	1	— 24
Sum of negative products			<u>— 38</u>
0.5 %	29	0	—
0.7 %	18	1	+ 18
0.9 %	7	2	+ 14
1.1 %	6	3	+ 18
Total frequencies	91		
Sum of positive products			+ 50

Average = $A + \frac{D \cdot C}{F}$. Where A = selected value, D = difference of products, C = value of class interval, and F = total frequencies.

In this case, average = $0.5 + \frac{(-38 + 50) 0.2}{91} = 0.53 \%$.

To obtain a fair average of normal samples, in some cases, the doubtful or adulterated extremes should be neglected, or the value of the middle sample of the series (median) be obtained. The calculation of the median of the above series is as follows :—

Half the number of frequencies	45.5
Subtract total of frequencies under 0.4	31.0
	<hr/>
Differences	14.5
	<hr/>
Frequency in next interval	29
Median = $0.4 + \frac{14.5 \times 0.2}{29}$	= 0.50.

As before, 0.2 is the value of the class interval.

COMPARATIVE ADULTERATION FIGURES

If 100 samples be bought from as many vendors, and one of them is adulterated, obviously the adulteration is 1.0 %. Suppose 98 genuine samples are bought as before, and *two* adulterated samples from the *one* vendor. The percentage of adulteration is doubled, the increase being due only to the method of sampling. It follows, therefore, that a comparison of different years may be fallacious if there is alteration in the average number of adulterated samples bought from vendors who sell them. Caution must also be exercised in comparing adulteration in different districts. The comparison of different classes of vendors is liable to the same error ; rarely is more than 1 sample taken from a milk shop, but 23 adulterated samples have been taken from one farmer.

To make comparisons more correct the writer has suggested calculating the number of vendors selling adulterated articles in a particular period, in relation to each 100 samples purchased in that period, and calling it the “ comparative adulteration figure.” The percentage of milk adulteration in Birmingham may be taken as an illustration. In one year it was 8.7 %, in the next 13.5 %—figures which suggest increased adulteration. Such a conclusion is incorrect, as the comparative adulteration figure for *each* year was 6.0. The explanation of this anomaly is that in the first year, on the average, 1.5 adulterated samples were taken from each vendor of such, and

in the second year the average number was increased to 2·3. The increase of 4·8 % was due *entirely* to sampling. In one year the percentage of adulteration of farmers' milks was more than twice as high as that of shop samples, the proportions being 8·5 % and 4·0 % respectively. Really, the shops were a little the worse, the comparative adulteration figures being 3·8 and 4·0 respectively.

During four years, 1908–11, another method of calculation was tried in Birmingham—the percentage of *vendors* selling adulterated samples. It was unsatisfactory and entailed much clerical work. Particulars of the results for the average of these years may be of interest.

Number of samples adulterated per 100 samples (percentage of adulteration)	8·7
Number of vendors selling adulterated samples per 100 samples (comparative adulteration figure)	4·2
Number of vendors selling adulterated samples per 100 vendors	10·0
Number of adulterated samples per vendor of such	1·8
Number of samples per vendor	2·1

STANDARD SAMPLING

The figure for the total percentage of adulteration depends on the nature of the articles bought and their relative liability to adulteration. In 1925, 8·3 % of the milk samples bought in England and Wales were adulterated, but only 1·5 % of the butter samples. An increase of the butter samples in place of milk samples would have reduced the apparent percentage of adulteration. Comparison of different districts should therefore be on the basis of the national proportion of samples purchased, which was : milk, 52, butter 9, spirits 2, drugs 5, other articles 32—total 100. The revised adulteration figure would be, the number of milks adulterated out of 52 samples, plus the number of butters adulterated out of 9 butters, plus, etc.

For two towns the percentages of adulteration given were 8·6 % and 4·6 % respectively, while with correction to standard sampling, the first was *increased* to 12·1 % and the second *decreased* to 3·1 %, making a great alteration in the relative figures of the two towns (Liverseege, *S.P.A.*, 1928, **53**, 86).

CHAPTER XI

MICROSCOPY OF STARCHES

Measurement. Sizes. Quantitative methods, lycopodium equivalents. Detection of starches in mixtures.

As such excellent illustrated works on microscopy are available as those of Winton ("Microscopy of Vegetable Foods"), Greenish ("Microscopical Examination of Food and Drugs"), and Wallis ("Analytical Microscopy"), the writer will only give the result of some personal investigations on common starches, chiefly in relation to the determination of their proportion in mixtures.

MEASUREMENT. In the quantitative analysis of mixtures a good variety of scales of measurement is important. In counts depending on the relative sizes of different starches it is much better to count grains which are contained, or not contained, between lines of the eyepiece micrometer, than to base counts on estimates of fractions of such divisions.

Difference in scale can be made by varying four things—(1) objectives, (2) eyepieces, (3) length of draw-tube, and (4) eyepiece squared micrometers. There is some difficulty with the latter, as microscope makers appear to be satisfied with offering only one micrometer eyepiece scale with an instrument.

Calibration of the scale is done most accurately by the method of coincidences. With a little care it will generally be found that a certain number of eyepiece divisions coincides with one or more stage micrometer divisions. Comparison is aided by the use of a microscope the stage of which can be mechanically moved and also rotated.

The Zappert counting chamber is a good standard, as it gives a choice of lengths of 5μ , 10μ and 100μ . For example, eighteen scale divisions were equal to four divisions of 100μ . The value of one scale division was therefore 22μ (μ = micron = 0.001 mm.).

The following calibration of a Leitz microscope in microns will illustrate the range that can be obtained, the last line uses the constant ratio between A and B as a test of the accuracy of the work :—

CALIBRATION OF COMBINATIONS OF LENSES

No. Objective	.	.	3	3	3	6	6	6	8	8	8
No. Eyepiece	.	.	0	2	5	0	2	5	0	2	5
Micrometer A	.	.	87	74	61	19	17	14	12	10	8
" B	.	.	33	29	24	7.7	6.7	5.5	4.7	4.0	3.3
A \times 0.4	.	.	35	30	24	7.6	6.8	5.6	4.8	4.0	3.2

SIZES. The following table gives lengths in microns of common starches, and the breadths of some oval ones :—

SIZES OF STARCHES

Starch.	Minimum.	Maximum.	Common Lengths.
Tous-les-mois . . .	16×14	119×84	Many 60–100 μ .
Potato . . .	5	91×70	Many 60–70 μ . very few under 5 μ .
Maranta Arrowroot	6	62×28	Many 30–40 μ .
Wheat . . .	3	76	A number over 40 μ , very few over 50 μ .
Barley . . .	2	38	Many 20–30 μ .
Sago . . .	10	60	Many 40–50 μ , few under 20 μ .
Tapioca . . .	3	28	Many 8–16 μ , damaged or flattened grains may be 45 μ .
Maize . . .	3	42	Many angular 14 μ and more.
Rice . . .	3	10	Many 7–8 μ , very few under 4 μ .
Oat . . .	3	10	Many 6–8 μ .

QUANTITATIVE METHODS. LYCOPodium EQUIVALENTS.

The following methods have been tried for the determination of the proportion of starches in mixtures :—(1) Comparison of the appearance under the microscope of an adulterated sample with others ; (2) counting the number of starch grains ; (3) determining the ratio of grains considered to be characteristic of the article and the adulterant ; and (4) determining the “ lycopodium equivalent,” which is the ratio of added lycopodium cells to total starch grains or those of a particular size. Examples of these methods will now be given.

(1) Simple comparison of slides of an adulterated article with those containing known amounts of adulterant (cp. Cleaver, *Analyst*, 1877, 1, 188). This method is not capable of giving exact results, as anyone may find who makes slides containing 5 %, 10 %, 15 %, 20 % of an adulterant and then tries by observation to place them in the proper order.

(2) Comparison by counting the number of starch grains in a slide made with a definite quantity of substance, and comparing the counts with those of similar standard slides, will give better results than simple observation. 0.1 gm. of a sample of mustard adulterated with wheat flour was mixed with 10 ml. water. The average of ten fields on a slide was 16.2 starch grains, and of a second slide 14.8 grains. Similar standard slides of mustard with 10 % of adulteration gave 28.4 and 32.2, and with 20 % 48.7 grains. It will be seen that duplicates do not agree closely, and that the 10 % figures are more than half the 20 % one. Calculation from various combinations of these figures indicates 4.6–6.1 % of adulteration.

To obtain correct results by this method it is necessary that the films containing the starch should be of equal thicknesses, and

that the starch grains should be uniformly distributed. The appearance under the microscope of a mixture of a large and smaller starch suggests that the little starch grains play round the pillars of their big brothers which support the cover glass. Pressure on the cover glass may squeeze the smaller grains out of the slide.

The thickness of the slide may be made uniform by the use of a counting chamber which provides a support for the cover glass at a definite distance (0.1 mm.) above the slide such as the Zappert chamber. In practice it is better to use the areas given by the eyepiece micrometer, which are always distinct, than those ruled on the chamber, which are at times indistinct. The constant depth is the important advantage of the chamber. The drop used, applied by a pointed glass rod, or platinum loop, should not quite cover the glass slide.

By the use of the chamber the number of grains present in a gramme can be determined; calculate as follows:—

$$\text{Millions of grains per gramme} = \frac{10,000 a b}{(\mu)_2 \times c}$$

a = No. grains per field. b = No. ml. which contain 1 gm. (1,000 is a convenient volume). μ = length of side of square of eyepiece micrometer in microns. c = No. of squares in the field, which will usually be that included in the extreme lines of the eyepiece micrometer.

Counted in this way *tous-les-mois* starch had 20 millions of starch grains per gramme, cornflour (air-dry) 920 millions, and lycopodium 86 millions. Wallis ("Analytical Microscopy") found 84.6 millions in air-dry maize starch, and 94 millions in lycopodium.

(3) The determination of ratios between two sizes of starch grains avoids the error due to differences in the thickness of compared slides; between limits, the total number of grains does not matter, but only the ratio between them.

The proportion of grains 10-20 μ to those under 10 μ is much larger in maize than in wheat, and this fact was made use of in the examination of a sample of flour which was labelled "Guaranteed Absolutely Pure." On examination with a No. 8 objective and a No. 2 eyepiece, of the grains which were less than 20 μ , 8.7 % were between 10 μ and 20 μ . With pure wheat flour the figure was 3.2 %, while 4.75 % of maize gave 6.8 % of larger grains, and 10 % of maize 11.0 %.

The last three figures may be used as a test of the process. Using the extremes as standards, the middle term indicates 4.6 % of maize, while 4.75 % had been added.

$$\text{Maize} = \frac{(6.8 - 3.2) 10}{11.0 - 3.2} = 4.6 \%$$

The application of the standard results to the sample gives :—

$$\text{Maize} = \frac{(8.7 - 3.2) 3.75}{6.8 - 3.2} = 7.3 \%, \text{ or } \frac{(8.7 - 3.2) 10}{11.0 - 3.2} = 7.0 \%$$

A method given (p. 123) indicated 9 %. The sample was certified to be adulterated with 7 % of maize flour, and the vendor was fined £5.

(4) In order to avoid any uncertainty as to the quantity of microscopical material that is actually being observed, Wallis (*S.P.A.*, 1916, **41**, 357 ; *B.P. Conf.*, 1919, 413 ; *P.J.*, 1921, Jan. 22) has suggested the addition of lycopodium in a definite proportion to the material examined, and the determination of the ratio between the starch grains and added lycopodium spores.

The writer has found the method very useful, and has used it as follows :—Mix 0.1 gm. of the material, in fine powder, with 0.1 gm. of lycopodium, and a little olive oil, either with a spatula on a tile or in a mortar ; the emulsion is gradually transferred to a 20 ml. stoppered cylinder, rubbing up with more oil till the volume is about 20 ml. After thorough mixing a slide made from it is examined and the number of starch grains, and also lycopodium spores, counted in a number of fields, taken at random all over the slide. Wallis recommends the addition of castor oil to give greater viscosity in hot weather, and the use of mucilage of tragacanth in certain cases as suspending agents.

Instead of counting all starch grains it is often of advantage to use a squared micrometer eyepiece and count only grains over a certain size or between certain sizes, according to the sizes of the starches in the mixture.

The above equal proportions will not always yield suitable fields for counting, and the amount of lycopodium in 20 ml. may be usefully varied. With a $\frac{3}{8}$ -inch objective, and variation of the eyepiece and of the length of draw tube, 0.1 gm. of lycopodium gave 5–17 spores per field, which is a convenient number. With $\frac{1}{8}$ -inch, only 1–5 were counted ; numbers which are too small. On the other hand, 0.5 gm. of lycopodium per 20 ml. gave 7–19 spores per field with $\frac{1}{8}$ -inch, but with $\frac{3}{8}$ -inch, 50–100 spores per field, which is inconveniently large. Again, the amount of material taken may be varied, not only for the magnification used, but also according to the amount of starch present, and the proportion of the grains counted. The upper limit is probably about 0.5 gm. per 20 ml.

Liverseege and Parsons (*S.P.A.*, 1922, **47**, 430) have suggested the use of the term “Lycopodium Equivalent” for the constant obtained in the examination of starches ; its use avoids any difficulty in the calculation of different proportions of starch and lycopodium.

$$\text{Lycopodium equivalent} = \frac{\text{No. of starch grains} \times \text{weight of lycopodium} \times 100}{\text{No. of lycopodium spores} \times \text{weight of substance}}$$

When equal weights of substance and lycopodium are taken, the L.E. is the number of starch grains for every 100 lycopodium spores. The quantity of olive oil does not enter into the calculation.

As the weight of 100 spores is about 0.001 mgm., the L.E. is the number of starch grains in about 0.001 mgm. of the substance.

With all work of this kind uniformity of method is essential; with a squared micrometer eyepiece it is convenient to ignore all grains which have less than half of the grain on the squared part. Fields with doubtful grains are better entirely ignored, but if many fields have masses of grains the material was not sufficiently finely powdered and a fresh emulsion should be made. After an emulsion has been standing a number of hours very thorough shaking may be necessary before a new count is made. After use the emulsion may be filtered and the oil used again.

To avoid variation in the amount of moisture, starches are better dried in the water oven before use, but this should not be done with oily substances such as mustard.

The following are examples of the determination by different workers of the proportion of wheat flour in compound mustards.

Lycopodium spores 455, starch grains over 17μ in wheat flour 284, L.E. over $17\mu = 62.4$, a second result was 64.6. A mixture of this flour with an equal quantity of mustard gave 35.1 and 31.4. Using these values indicates 53 % of flour was found against 50 % added.

The average L.E. found by one worker (M.) for a compound mustard was 23.9, and another (L.) found 24.2. For a second sample (L.) obtained 29.1, and (B.) 28.8. Although these agreements are good, it is advisable that each worker should use his own constants as standards.

When the first standard differs considerably from the sample, the result should be used as the basis for the preparation of a second standard, so that the final comparison can be made on two similar counts.

The simple starch grains over 17μ in a sample of pig meal made up of oat and barley meals gave an L.E. of 63. For comparison standards of 5 % and 50 % of barley were made, the first gave an L.E. of 12 and the second 70. If only barley grains were counted, one figure would be ten times the other; as that was not the case the following method of calculation is more correct:—

$$\text{Percentage of barley over 5 \%} = \frac{(63 - 12)(50 - 5)}{70 - 12} = 39.6 \%$$

Adding the 5 % gives 44.6 % of barley in the pig meal.

DETECTION OF STARCHES IN MIXTURES. In the examination of a suspected sample for a particular starch there is a great risk of self-deceit. The hunt for a particular type of starch

may become so keen that the sense of proportion is lost and one or two grains may be taken as a proof of the presence of an adulterant, without an equally keen examination of an authentic sample. Descriptions and illustrations are useful, but the important thing in the search for a small proportion of adulteration is that the acquaintance with a particular starch shall not be casual, but intimate. This work is often tedious and tantalising, but necessary to obtain confidence in one's results.

It should also be remembered that commercial flours need not be of 100 % purity to be passed as genuine. Wheat, barley, and oats are all cultivated in this country, and while the removal of foreign seeds from grain is usually well done, the presence of small proportions of foreign grain may be due to accident and not to deliberate adulteration.

When possible it is better to make comparisons with starch taken directly from the grain, but with small starches staining with iodine may be advisable to prevent mistaking aleurone grains for starch. Owing to the method of preparation of commercial starches they may differ in range of size from that taken from the grain directly.

It is often useful to watch starches while the cover glass is being gently moved; this will distinguish between spheroidal and flattened grains. Some observations by this method are given below.

Tous-les-mois. Owing to the large size of this starch its substitution for arrowroot is easily detected.

Potato. The larger grains are ovate, but a number are circular. Occasionally twin grains are present; one grain having a total length of 39μ and a breadth of 22μ might have been mistaken for tapioca. The L.E. of grains over 19μ in a sample of potato starch was 44. The grains are not flattened, the hilum is annular, or occasionally a star or slit, and polarised light usually shows the cross at the small end of the grain.

Sago. The larger size of these truncated grains readily distinguishes them from tapioca. The outer grains of masses of both sago and tapioca are distorted by heat, but by cutting open, unaltered starch grains may often be found.

Tapioca. There are three classes of truncated starch grains. From the flat surface the sides may: (1) Slightly converge or diverge, and these grains may be called muller shaped. (2) The lines may converge and then diverge, giving an urn-shaped grain. (3) The sides may be at right angles to the flat surface and parallel for perhaps two-thirds of the grain. "Tombstones" appears to be a suitable name for such grains, particularly as at the rounded part a slit hilum takes the place of an inscription. Tapioca differs from sweet potato in having no "tombstones." The grains are

not flat except at the base, upon which they often stand and appear circular in outline. Some have angular facets like dice. Twin or triple aggregates are present. The hilum is usually in the middle of the grain, in the form of a dot, pit, star, wings, but is sometimes invisible. Mounted in cedar oil, it does not show as a black spot. The L.E. of grains 9.5μ and less is about 300, and for grains 10.4μ and less about 610.

Sweet Potato. *Ipomoea Batatas*. Stubbs (*S.P.A.*, 1926, 51, 400) has described this starch. It has many angular and faceted grains like dice, and globular grains $10-25\mu$. It differs from tapioca in having "tombstone" grains. The hilum is absent in many grains, and occurs as dot, slit or star in others. In "tombstones" it is a slit about one quarter of the way down; in others it is central.

Maranta Arrowroot. The grains are oval (both ends alike), ovate, pear or oyster shape. Only small grains (10μ) are circular. Some grains ($20-30\mu$) are almost squares. The length is rarely more than one and a half times the breadth. The hilum, when visible, may resemble a star, cross, wings, circular dot or pit, or a slit which is sometimes parallel to the length of the grain. The hilum as shown by polarised light varies from central to one quarter from either end. Aggregates are absent. Bermuda is rather larger than other kinds, many grains are over 40μ . There is a nipple-shaped projection on very few grains.

In the table below results are given for three adulterated arrowroots in which the percentages of tapioca were determined by lycopodium equivalents. Five samples free from tapioca, probably all St. Vincent, gave L.E. varying from 17-25 for over 34.4μ , and 26-30 for those over 30.4μ , the means being 20 and 33 respectively. Owing to these variations it is more satisfactory to calculate from the tapioca L.E. given above:—

MIXTURES OF ARROWROOT AND TAPIOCA

					STANDARDS.		SAMPLES.		
					Arrowroot.	Tapioca	1.	2.	3
LYCOPodium EQUIVALENTS.									
A.	Over 34.4μ	.	.	.	20	—	8	9	18
B.	Over 30.4μ	.	.	.	33	—	15	18	23
C.	Under 10.5μ	.	.	.	—	610	312	412	174
D.	Under 9.6μ	.	.	.	—	300	—	—	116
ARROWROOT %									
Using A	—	—	40	45	90
Using B	—	—	45	55	70
TAPIOCA %									
From A, by difference	—	—	60	55	10
From B, by difference	—	—	55	45	30
Using C	—	—	51	68	29
Using D	—	—	—	—	39

Wheat and Barley. These starches are very similar in appearance. Each is flattened, wheat being probably triangular in section. The large grains of each are circular, or nearly so. The small grains of wheat are round or oval, a few are angular, and some resemble tapioca. Not many of the small barley grains are round; many are angular or have a flattened side. The hilum of wheat is rare in large grains; when barley grains are on edge the hilum looks long. With $\frac{2}{3}$ -inch objective and polarised light, only a few large grains of wheat show a faint cross, but barley has many crosses.

The L.E. for grains 17μ and over is about 80 for dried wheat flour, and that of a sample of dried wheat starch was about 200. The L.E. for grains over 5.3μ for undried wheat flour was about 200.

In the following table is given the proportion of different sizes of starch grains in wheat starch, in starch taken directly from wheat grains, and in barley meal. It is based on counts made by several workers in my laboratory. They only include about half the total grains, those under 5μ are difficult to count so as to get concordant results. A paper by Wallis (B.P. Conf., 1922, 373) on the subject should be consulted.

SIZES OF STARCH GRAINS IN WHEAT AND BARLEY

Percentage of Grains.	Wheat Starch.	Wheat Grain.	Barley Meal.
5μ	59	77	40
10μ	14	12	16
15μ	11	4	21
20μ	16	7	23
	<hr/> 100 <hr/>	<hr/> 100 <hr/>	<hr/> 100 <hr/>

Wheat starch exceeds wheat grain in the proportion of starch grains over 15μ , and barley meal has still more. The table should be a guide as to the size of the grains best to be counted in mixtures containing other starches.

Maize. The grains are almost always polyhedral and not flattened. The angles are usually rounded. The hilum is a star, slit or circle, but is often invisible with $\frac{1}{8}$ -inch objective. Very few grains give a cross with polarised light and $\frac{2}{3}$ -inch objective, and none with $\frac{1}{8}$ -inch.

The range of size is as follows : grains under 5μ are not included : 5μ - 38 %, 10μ - 44 %, 15μ - 14 %, 20μ - 4 %.

When mounted in oil of cloves, or cedar, the hila of wheat starch are practically invisible, while those of maize appear as black dots (Wilson, *Analyst*, 1900, **25**, 316). With a "B" eyepiece and $\frac{1}{8}$ -inch objective 2 % of maize is readily detected. The adulterated wheat flour, mentioned on p. 118, had no hila visible in 144 grains over 14μ ; and 19 grains over 14μ showed black hila, or 13.2 % of the grains observed. In the same conditions 14.5 % of the grains in

a 10 % mixture were indicated. The relation between the figures shows that 9 % of maize was present in the adulterated sample.

Maize starch is less acted on by alkali than wheat, as pointed out by E. W. T. Jones (*Analyst*, 1900, **25**, 317). The following is a modification of his method. Shake 1 gm. of flour thoroughly with 10 ml. of water, then add 15 ml. E/4 NaHO, and again shake well. Allow to stand for about two hours, and dilute with 75 ml. water, to stop the action. Examine the emulsion, or deposit after standing, with $\frac{2}{3}$ -inch or $\frac{1}{8}$ -inch objective. Genuine wheat flour will have particles about 2μ , with a few up to 5μ , but maize will have angular grains 16–20 μ with stellate hila. Embrey has devised a quantitative method (*S.P.A.*, 1900, **25**, 315).

Rice. Most grains are irregular in shape with sharply defined angles. It may be in masses or compound grains. The range in size of a ground rice grain was 2.5 μ – 14 %, 5.0 μ – 62 %, 7.5 μ – 44 %. Iodine was used to stain the grains. Rice may be detected in pepper by observing if there are grains more than 5 μ .

Mounted in oil of cloves and examined with $\frac{1}{8}$ -inch objective, some grains showed darkening in the middle, but not so much as maize. Rice was more resistant to weak alkali than maize; a large number of unaltered starch grains remained after standing two months.

To detect rice in wheat flour, Bellier (*Analyst*, 1907, **32**, 386) has suggested using an aqueous solution of 5 w/v KHO, and 15 v/v of glycerin, as a reagent. 0.5 ml. of it is added to about 0.2 gm. of the flour in a beaker; it is well stirred and examined under the microscope after thirty minutes. Wheat flour becomes transparent, while rice is white and little altered in appearance. 1 % of rice can be detected by using $\frac{2}{3}$ -inch objective and confirmed under a higher power. 1 % and 2 % were similar, but could be distinguished from 5 % and 10 % (cp. Wagenaar, *Analyst*, 1928, **53**, 100).

Oat. The grains are similar to rice, but some are round. Compound grains are common. The range in size was found to be—2.5–4 μ —20 %, 5–7 μ —50 %, 7–10 μ —20 %, 10–13 μ —10 %.

White, in a paper on the detection of maize in oatmeal (*S.P.A.*, 1895, **20**, 31), states that an occasional oat grain will show a cross with polarised light, but the crosses are very much smaller and not so well defined as those of maize.

CHAPTER XII

STARCHY FOODS

Bread. Cake, sponge-cake, etc. Flour. Self-raising flour. Pudding powder. Semolina, macaroni, vermicelli. Rice. Pearl barley. Oatmeal. Rolled oats. Cornflour. Indian meal. Sago. Tapioca. Arrowroot.

BREAD

IN 1911 Hamill made a long report to the L.G.B. "On the nutritive value of bread made from different varieties of wheat flour" (Food Report, No. 14). A Ministry of Food Order in 1917 stated that on the average a sack of flour (280 lb.) produced eighty-nine quartern loaves.

As a rule the crumb of bread only is analysed; the difference in composition is shown by an analysis made in 1896. The crust contained 17.5 % of moisture, and 1.40 % of ash; the figures for the crumb were 40.6 % and 0.96 % respectively. Hertwig and Bailey (*Analyst*, 1926, **51**, 38) give a method for determination of the moisture in an entire loaf.

In 1917 the Food Controller required the addition of other cereals to wheat flour. The following average analyses of Birmingham samples indicate that there was little difference in the two varieties :—

ANALYSES OF BREAD CRUMB

	Moisture. %	Fat. %	Nitrogen. %	Ash. %	Alkalinity of Ash. N.v/w.
White bread.	41.9	0.2	1.20	1.0	4.5
War bread .	41.4	0.4	1.13	0.9	7.3

The following analyses relate to the crumb of ordinary white bread. The ash of twenty-one samples varied from 0.83 % to 1.27 %, the average being 0.9 %. The average percentage of P_2O_5 in fifteen samples was 0.15, the range being 0.11–0.25 %.

MOISTURE IN BREAD CRUMB (Thirty-three samples)

Percentage of moisture	. 37.8–	40.0–	41.0–	42.0–	43.0–44.5	Total
Percentage of samples	. 9	40	30	15	6	100

The early reports of the L.G.B. on the Sale of Food and Drugs Acts have many references to the presence of alum in bread. Of the 6,543 samples examined 1877–82, 432, or 6.6 %, were adulterated, chiefly with alum. In 1880 one sample contained 1,305 grains of alum per 4-lb. loaf! During 1900–13, the proportion of adulteration

fell to 0.4 %, and to 0.2 % during 1920-31. In Birmingham, 2,000 samples were examined 1884-1931, and only two, in 1884 and 1889, contained even small quantities of alum.

Three samples of "*Bread improver*" were analysed. One was a mixture of about equal parts of acid calcium phosphate and wheat flour. Another was an acid calcium phosphate containing calcium sulphate, and claimed that its use would yield 32 lb. extra bread per sack of flour, which is equal to the addition of about 8 % of water to the bread. If the directions for use were followed, the "improver" would add about 0.4 % of ash to the bread, of which about 0.2 % would be P_2O_5 . The third sample was a war-time local make. It claimed to produce more bread of a better quality, to save yeast, and to add moisture preventing the loss of weight of the bread on the second day. About half the article was rice and potato starch, and about one-third of it was Epsom salt, the remainder being moisture and mineral matter containing a little phosphate. The price charged was 8*d.* per lb. Loaves were examined which had been made with the "improver" and others for comparison. The claim for increased volume was disproved by the sp. gr. which was 0.50, while normal loaves were 0.47. Between eighteen hours after baking, when the loaves were received, and eighty-nine hours, each set of loaves lost 4.7 % of weight. During the same period normal cut loaves lost 6.5 % and treated 6.4 %. The crumb of the loaves analysed eighty-nine hours after baking contained 44.2 % of moisture, and the "improved" bread 44.1 %. The normal loaves had 1.7 % of ash, and the others 2.0 %. The treated loaves tasted slightly salt. The legal definition of "food," as an "article which ordinarily enters into or is used in the composition or preparation of human food," can hardly be held to include bread "improvers," and the police solicitor considered it inadvisable to prosecute for obtaining money under false pretences. The researches of Wood and Hardy (*Proceedings of the Royal Society*, 1909) show that Epsom salt increases the coherence ("strength") of gluten in the presence of acid; the effect was, however, only about one-third of that of common salt.

Another war-time product of the same firm may be mentioned. It made great claims as a substitute for grease on bread tins, and was sold at 1*s.* per lb. It consisted of fine sawdust!

ANALYSIS. Maize. A loaf was made in which 20 % of the flour used was maize. The bread, on microscopical examination mounted in water or oil of cloves, showed only a few characteristic starch grains. Fricke and Luning (*Analyst*, 1923, **48**, 331) claim that 5 % of maize flour can be detected by testing for characteristic maize proteins.

Mineral Matter. According to Curtel (*Analyst*, 1910, **35**, 398), these can be detected by sedimentation as with flour (p. 133), if

the bread be previously boiled with the carbon-tetrachloride. The determination of *alum* has been given previously (p. 93).

PROSECUTIONS. *Wednesbury.* Alum. It had been used to make presentable loaves with damaged flour. Fine £25 (*Analyst*, 1877, 1, 117).

Pendleton. Alum 16 grains per 4-lb. loaf. The baker stated that, without his knowledge, his workmen had added alum to save trouble, as the flour worked up more easily with it. Fine £10 (*Analyst*, 1879, 4, 98).

Salford. Alum 40 grains per 4-lb. loaf. Fine £4 (*Analyst*, 1879, 4, 138).

Nottingham. Alum, to the prejudice of the purchaser. The baker denied adding alum, and said that if it were there it came from the flour. The justices dismissed the case, holding he was not liable, as he did not know alum was present. This decision was reversed by the High Court (*Bells v. Armstead*), holding that an offence had been committed, though the seller did not know the article was incorrect (*Analyst*, 1888, 13, 119).

Wolverhampton. Alum 57 grains per 4-lb. loaf. Fine £1 (*F. & S.*, 1893, Aug. 19).

Nottingham. Alum 18 grains per 4-lb. loaf. The Medical Officer of Health stated that persons who took the bread regularly would undoubtedly suffer ill effects. Fine £1 (*B.F.J.*, 1907, 172).

Hexham. Arsenic $\frac{1}{10}$ grain per lb. The baker was unable to explain its presence, phosphates had not been used, and arsenic had not been detected in the flour, yeast, etc. Fine £5 (*Grocer*, 1917, Feb. 24).

Grimsby. Calcium sulphate 11·8 and 16·8 grains per lb. respectively; it was due to an impure acid calcium phosphate used as a bread improver. The Medical Officer of Health stated, that, in his opinion, calcium phosphate and sulphate were injurious, especially to children. As they were the first cases of such adulteration, each defendant was fined 1s. only (*Grocer*, 1917, Sept. 1; *B.F.J.*, 1917, 178).

CAKE, SPONGE CAKE, ETC.

Sponge cakes which are largely used for children and invalids are made from about equal parts of flour, sugar and eggs. A sample contained 9·6 % protein, 6·2 % fat, 0·9 % ash, with starch and 15·1 % moisture. Cake contains much less eggs; in a common cake mixture there may be about 1 lb. of eggs in 7 lb. of cake. In 1889 two Birmingham currant lunch cakes contained 189 grains alum per 4 lb.

In 1921 the Ministry of Health sent out a circular calling attention

to the fact that liquid eggs containing a notable amount of boric acid were being used in making cakes.

The analysis of a number of samples bought in Birmingham in 1921 showed that 95 % of the fruit, etc., cakes contained boric acid, and 74 % of the sponge cakes, one of the latter having 59 grains of boric acid per lb. ! Other samples containing 35 grains per lb. were found to be made with liquid eggs containing 1·8 % of boric acid ; the other constituents were free from it. In some cases two sponge cakes contained a dose of boric acid ! On the instructions of the Public Health Committee, the Medical Officer of Health sent out a circular to the bakers and confectioners of the city. In the next year there was a great improvement, but one confectioner was fined £10. In 1923 the Ministry of Health (Circular 381, *Analyst*, 1923, 48, 177) announced an agreement with the trade limiting the use of boric acid, and the Preservative Regulations prohibited entirely its use in 1928.

The following table shows the progressive improvement both in the percentage of samples free from boric acid, and also in the proportion used :—

BORIC ACID.	SPONGE CAKES, ROLLS, SANDWICHES.				FRUIT, MADEIRA, ETC., CAKES.			
	1921.	1922-5.	1926-7.	1928-30.	1921.	1922-5.	1926-7.	1928-30.
None . . .	26	71	96	100	5	14	52	100
1 to 7 grains per lb..	4	0	2	—	75	79	38	—
8 and over „ .	70	29	2	—	20	7	10	—
	100	100	100	100	100	100	100	100
Maximum „ .	59	42	13		22	10	17	0

In connection with the Cake and Pastry Order, 1917, after a conference of analysts, a method for the determination of sugar in baked articles was proposed by the Government Chemist (*Analyst*, 1917, 42, 293).

According to the Ministry of Health Reports, 10·1 % of the samples of cake and biscuits analysed in England and Wales, 1922-30, were adulterated.

Below are given particulars of prosecutions owing to the presence of alum or boric acid. The L.G.B. Report for 1880 mentions two unusual adulterations—oatcakes with 10 % of chalk, and “ Baker’s Mixture ” having a considerable amount of arsenic, due to impure chemicals.

PROSECUTIONS FOR CURRANT BREAD. *Pontypridd.* Alum 25 grains per 4-lb. loaf. The Medical Officer of Health said that the alum would upset the mucous membrane of the alimentary canal, and be prejudicial to health. The Government Chemist refused to analyse the third sample, as it was not representative, consisting of 3 oz. only of outside parings and scrapings. The case was dismissed owing to informalities (*F. & S.*, 1894, April 7, 28).

Wolverhampton. Alum 210 grains per 4-lb. loaf. An egg powder which contained 37·4 % of alum had been used in preparing the bread. The vendor was fined £3 for selling an article injurious to health (*F. & S.*, 1894, July 21).

PROSECUTIONS FOR GINGERBREAD. *Wolverhampton.* Alum 46 and 72 grains per lb. The vendor was fined 33s. 6d. Two other vendors whose gingerbread contained 70 and 75 grains of alum per lb. were each fined 19s. 6d. (*F. & S.*, 1898, July 16).

PROSECUTION FOR BROWN BISCUITS. *Pontypridd.* Alumina compound derived from the addition of alum, and equivalent to 80 grains of alum per lb., which admixture was injurious to health. Fine £5. (*F. & S.*, 1895, Oct. 19).

PROSECUTION FOR SPONGE SANDWICH. *Torquay.* Boric acid 35 grains per lb. Defendant had used liquid eggs innocently and was ordered to pay costs (*Grocer*, 1923, June 9; *B.F.J.*, 1923, 60).

PROSECUTION FOR FAIRY CAKES. *London, South-Western.* Boron preservative, equivalent to 24·6 grains of crystallised boric acid per lb. After contradictory medical evidence the case was dismissed (*Grocer*, 1925, March 7, 21, 28; *B.F.J.*, 1925, 36, 46).

PROSECUTION FOR SPONGE FINGERS. *Holt.* Boric acid 4·2 grains per lb. They had been made with a mixture of fresh eggs and preserved liquid egg yolk. Fine 10s. (*B.F.J.*, 1926, 49).

PROSECUTIONS FOR SPONGE CAKES. *Birmingham, 1922.* Boric acid 33 grains per lb., and injurious to health. The defendant, who admitted that he read the trade papers, failed to convince the magistrates "that he did not know, and could not with reasonable diligence have ascertained," the presence of boric acid in the liquid eggs he had used, and was fined £10. He gave notice of appeal to Quarter Sessions, but subsequently withdrew it.

Stoke-on-Trent. Boric acid 5·4 grains per lb. Liquid eggs had been used instead of shell eggs. Fine 21s. and 21s. costs (*Grocer*, 1927, Feb. 5; *B.F.J.*, 1927, 30).

FLOUR

Although by dictionary usage the word "flour" may mean the soft white powder of any substance, if "flour" is asked for at a baker's or grocer's shop, only wheat flour should be supplied. The term "wheaten flour" is said to be obsolete and liable to suggest to a vendor that wheatmeal containing bran is required.

A report to the L.G.B. made by Hamill (Food Report, No. 14, "On the nutritive value of bread made from different varieties of wheat flour") gives details of the structure of the wheat grain, and the practice of milling. He also gives numerous analyses, taken

from American sources, of various wheats and the grades of flour made from them.

Water has been sprayed on flour (Hamill, Food Report, No. 12), and it has been suggested (*B.F.J.*, 1927, 92) that flour should not contain more than 15 % of moisture.

MOISTURE IN FLOUR (Fifty-six samples)

Percentage of moisture	10.1-	11.0-	12.0-	13.0-14.1	Total
Percentage of samples	9	27	46	18	100

The next table gives the range of the amount of ash, divided into three periods. In March, 1917, the war-time Food Controller made an order requiring the mixture of other cereals with wheat flour :—

ASH IN FLOUR

Percentage of Ash.	1896-1916.	1917-1918.	1919-1931.
0.28-	12	0	17
0.4-	67	7	69
0.6-	19	34	12
0.8-	2	29	2
1.0-1.37	0	30	0
	<hr/>	<hr/>	<hr/>
	100	100	100
Number of samples	543	86	391

During the first and third period only a few samples were over 0.8 % of ash, the highest being 0.97 %. In the war period more than half the samples exceeded 0.8 %.

The amount of phosphoric anhydride (P_2O_5) varied from 0.1 % to 0.56 %; about 60 % of the samples containing between 0.2 % and 0.4 %. About 50 % to 60 % of the total ash was P_2O_5 . Flour yields an ash alkaline to methyl orange, 2 to 4 N.v/w. Flour contains very little sulphate, the average of eighteen samples being 0.02 %.

Sixteen samples contained from 1.67 % to 1.94 % of nitrogen, the average being 1.87 %. The average of five samples examined in 1922 was : fat 1.2 %, spirit extract 1.8 %, and water extract 10.6 %, of which 0.4 % was ash.

In recent years a great deal of attention has been given to the bleaching of flour and the use of "improvers." About 1905 a process of bleaching by nitrogen peroxide was applied to English flour, producing an improvement in the whiteness of the lower grades and thus increasing their commercial value by deceiving the purchaser. In 1911, Hamill made a report to the L.G.B. on bleaching and the addition of "improvers" (Food Report, No. 12). He considered that it would be unwise to conclude that the use of flour bleached with nitrogen peroxide was devoid of risk to the consumer. He regarded with considerable apprehension the addition of foreign substances of doubtful utility to flour, and pointed

out that the addition of phosphates did not increase the natural organic phosphorus of the flour.

At the same time a report by Monier-Williams gave experiments on the chemical changes produced in flour by bleaching (Food Report, No. 12). In 1912 he reported that the colouring matter of flour was carotin, or very similar substances, and considered it to be extremely improbable that unbleached flour by storage under ordinary conditions will show more than 1.5 to 2.0 parts of sodium nitrite per million on testing (Food Report, No. 19). In 1927, Kent-Jones and Herd emphasised the great public demand for clean, bright and white flour, and gave a method for determining the colour of flour (*S.P.A.*, 1927, 52, 443). They stated that the colour is due to traces of bran as well as to carotin, and that carefully milled flour has very little bran. Bleaching will destroy the carotin but have little effect on the bran. Thompson has also discussed the difference between the natural and artificial bleaching of flour (*S.P.A.*, 1914, 39, 522).

A prosecution at Hamilton in 1911 for nitrites in flour, and one at Hull in 1915 for persulphate, were both dismissed after lengthy hearings.

Treatment of flour was not desired by bakers. In 1911 an expert committee appointed by the Council of the National Association recommended that the baking trade should insist on a guarantee that "flour was unbleached and untreated, and free from any added substance whatever."

In 1924 the question of the treatment of flour by chemical substances was referred by the Ministry of Health to a Departmental Committee, and a Report was issued in 1927.

The Report stated that chemical substances added to flour made it mature more rapidly both in whiteness and "strength." As flours from various wheats differ considerably in "strength," the use of "improvers" makes blended flours more uniform. Evidence was given that flour heated in a particular way could be used as an improver instead of chemicals.

The Committee considered "that flour should be the product of the milling of wheat without the addition of any foreign substances," but "that in the first instance it should suffice to limit the use to those which appear least open to objection," such as nitrogen trioxide and persulphates for bleaching, and acid calcium and ammonium phosphates for "strength." The use of chlorine, nitrogen trichloride, and benzoyl peroxide are condemned. Unfortunately, the analytical distinction between flour containing the latter, and that containing persulphates, is very difficult, or impossible. The Committee held that the claim that improvers produced a damper bread was not proved.

The Report was not followed by any Parliamentary action, but

in 1929, under the Agricultural Produce (Grading and Marking) Act, 1928, standards were made for National Mark flour. They required such flour to be unbleached by artificial means, to be free from all added chemical substances, and to yield not more than 0.55 % of ash by ignition in a muffle furnace.

In the five years commencing 1910, 57 % of the Birmingham samples of flour contained from 2 to 10 parts of sodium nitrite per million as the result of such bleaching. Before 1905 flour was sold in its natural state, but gradually bleached flour was sold in the place of natural flour, without any notice being given to the consumer of the substitution that had been made. In 1915-20 the proportion fell to 39 %, and in 1921-7 it fell to 17 %. In some cases persulphate or benzoyl peroxide was present as well as nitrite. During the years 1924-31, 28 % of the samples of flour contained persulphate or peroxide. In 1927-9 twenty-four samples were tested for sulphur dioxide, but it was not detected.

For many years alum was the chief adulterant of flour, and the Bread Acts of 1822 and 1836 were passed to prohibit its use. Alum was added to produce a whiter and better-looking loaf from inferior flour.

Alum was not detected in any of the 1,740 samples of flour examined in Birmingham from 1873 to 1931. In 1913 a sample was condemned for the presence of 0.5 % of calcium superphosphate; one sample was self-raising flour, probably sold as "flour" by accident. In 1915 ten samples were adulterated with maize, some of them being marked "Biscuit flour," which is the highest quality of flour. One sample was marked "Plain pastry flour" in large letters, and at the bottom of the bag, where it would not be noticed, there was in small type: "A blend of the finest English, Hungarian and maize flour." Such a label is not intended for the information of the purchaser, but as a protection against the Food Adulteration Acts. Such a label would be ineffective if, at a prosecution, the magistrates considered the addition was "intended fraudulently to increase its bulk . . . or to conceal its inferior quality." A vendor of such a flour admitted to the writer that maize flour was cheaper than wheat flour, but said he should use the former even if it were dearer, owing to the great improvement it made in the whiteness of the flour. In 1917-8 many samples contained maize by order of the Food Controller.

Of the samples of flour examined in England and Wales during 1877-9, 3.4 % were condemned, chiefly owing to the presence of alum. By 1900-13 the proportion had fallen to 1.6 %, and during 1919-30 it was 1.1 %.

Analysis. Potassium Persulphate and Benzoyl Peroxide. (1) After Rothenfusser (*Analyst*, 1909, **34**, 37) and Hinks (*S.P.A.*, 1912, **37**, 91). Take about 5 gm. of flour in a flat porcelain dish,

make into a paste with water and pour over it a little 4 % alcoholic solution of benzidine. There should be no blue specks.

(2) After Miller (*J.S.C.I.*, 1924, 239 T). Make about 5 gm. flour into a paste with a 10 % solution of KI, leave a few minutes and put dish on water bath for five minutes. Persulphate shows without heating, and peroxide by dark spots on the under side of the cake after cooling. Particles of husk may possibly be mistaken for the reaction.

(3) Rothenfusser advocates the use of *p*-diamino-diphenylamine (*Analyst*, 1925, **50**, 243) and Kirchoff recommends titanium sulphuric acid (*Analyst*, 1925, **50**, 406).

Nitrites. Put 7 ml. of nitrite-free water into uniform test tubes $5 \times \frac{5}{8}$ inch, add 2.5 gm. of flour and shake at once. Add 2 ml. of the Greiss-Ilosvay reagent, shake and allow to stand till the flour has settled, or longer if necessary. Prepare standards of nitrite-free flour with the addition of nitrite solution containing 5 parts of NaNO_2 per million, with water to make 7 ml. Each ml. used to match the colour given by a flour indicates 2 parts of NaNO_2 per million. The reagent is made by heating 0.1 gm. α -naphthylamine with 20 ml. glacial acetic acid and diluting with 130 ml. E.H $\bar{\text{A}}$; and mixing with 0.5 gm. sulphanilic acid dissolved in 150 ml. E.H $\bar{\text{A}}$ (*Analyst*, 1906, **31**, 300).

Superphosphate, Alum, Persulphate, or other Mineral Matter. Shake 10 gm. or more of flour with dry chloroform or carbon tetrachloride in a large separator, allow to settle over night, run the mineral additions through the tap, and apply appropriate tests. Flour will yield no phosphate (Curtel, *Analyst*, 1910, **35**, 398).

Sulphate. See General Methods, and also Thomson (*S.P.A.*, 1914, **39**, 526), who considers that naturally about one-fortieth of the mineral matter in flour is SO_3 . Cripps and Wright pointed out that in the presence of acid phosphate, sulphate is lost on ignition (*S.P.A.*, 1914, **39**, 429).

Acidity. Mix 10 gm. of flour with 100 ml. of water, add methyl red and titrate with N/10 NaHO. Normal flours will not exceed 0.2 N.v/w. An old sample gave 5.8 N.v/w in 1916 and 7.4 in 1929. Phenol phthalein gives much higher results. See Thompson (*S.P.A.*, 1914, **39**, 519) and Marion (*Analyst*, 1909, **34**, 354), who found that acidity increased during the first year, and then fell slowly and gradually.

Gluten. The determination is difficult; see Kent-Jones and Herd (*S.P.A.*, 1927, **52**, 439).

Water and Spirit Extracts. To 10 gm. flour in a flask add 100 ml. water and shake well. Repeat shaking at intervals and filter after twenty-four hours. Evaporate 50 ml. to dryness and dry in oven. After weighing the ash should be determined. The spirit extract is determined similarly.

Chlorine. For the detection of chlorine in flour see Kent-Jones and Herd (*J.S.C.I.*, 1930, 223 T; *Analyst*, 1930, **55**, 394).

PROSECUTIONS FOR FLOUR. *Baslow.* Alum 150 grains per 4-lb. loaf. The sample, taken in a mill, was marked "Flour-fourths," and it was contended that it was not used for human food, but for pigs. The vendor did not know how the alum got in. Case dismissed (*Analyst*, 1879, **4**, 235).

Eckington. Alum, 24 grains per 4 lb. The Public Analyst stated that the alumina present corresponded to 30 grains of alum per 4 lb., and that he had deducted 6 grains for that naturally present. By treating the flour with chloroform, he had proved the presence of alum. An analyst for the defendant stated that he had found alumina equivalent to about 23 grains of alum, and also 11 grains of silica per 4 lb.—quantities which were sometimes found in pure flour. Subsequently the Government analysts reported the presence of 18 grains of alum per 4 lb. The case was withdrawn, owing to the death of the defendant (*Analyst*, 1880, **5**, 71, 85).

Retford. A miller was prosecuted under the 1836 Bread Act for having in his possession alum intended to be used for the adulteration of flour. A servant gave evidence that he was told to add three handfuls of alum to each sack of flour. Fine £10 (*Analyst*, 1880, **5**, 84).

Southampton. Ground maize 35 %. The Public Analyst stated that he should understand by "flour" ground and bolted wheat, without maize in it. The magistrates, relying on their general knowledge and a dictionary definition, dismissed the case, as the Inspector had not asked for "wheaten flour." They refused to state a case for appeal to the Queen's Bench, as they considered no point of law was involved. Subsequently that Court granted a rule ordering the magistrates to state a case. One of the justices remarked that the dictionary did not support the contention that flour was to consist of two different kinds of grain (*R. v. Perkins and Mitchell, B.F.J.*, 1899, 25, 84).

Brentford. Bran 12 % in wheaten flour. After the sale was completed the Inspector saw the words "Wheat Meal" on the bag. Fine £1 and costs. An appeal to the Sessions followed, when the Public Analyst said flour should be the fine portion of wheat, free from husk. A member of the London Corn Exchange contended that flour and meal were synonymous terms, and that "wheaten flour" was an antiquated term, confusing to the trade. The case was settled on the undertaking by the defendants not to sell the article as "flour" or "wheat flour" (*B.F.J.*, 1902, 182, 251).

Handsworth. Acid phosphate of lime 1.37 %, which contained over 50 % of sulphate of lime. The bag was marked "Plain Flour." Fine 1s. (*B.F.J.*, 1910, 71).

Hamilton. Nitrites (stated as nitrite of soda) 3.43, or thereby,

parts per million, which was in excess of that contained in genuine flour. After a hearing which lasted eight days, the Sheriff concluded that the offence was not proved. He considered the flour had been bleached by peroxide of nitrogen, although evidence was given for the defence that unbleached flour might possibly obtain such an amount from the atmosphere. Evidence was given for the prosecution that large doses of peroxide of nitrogen had a deleterious effect on the composition of flour, but the Sheriff thought that small quantities, while affecting the colour, might have no prejudicial effect on its valuable properties, and he accepted the evidence for the defence that commercially bleached flours were unaltered in their essential characteristics. He considered that undue stress had been laid on the question of whiteness, and remarked that no witness had given evidence that he had been deceived by a second grade bleached flour being supplied to him as first grade. If the nature, substance and quality of the flour were not altered, the alteration of colour by bleaching, to suit the taste of the consumer, was not illegal. An appeal was entered, but withdrawn, as the County Council was advised that the practice of bleaching flour could not be dealt with under the Food and Drugs Acts (*Grocer*, 1911, Dec. 16, 23; 1912, March 30, June 1, 8, July 13, Aug. 3; *B.F.J.*, 1912, 11, 58, 106, 131, 152).

Hull. Potassium persulphate 6.3 parts per 100,000. The sample had been taken in course of delivery from a miller. The case occupied four days and fourteen witnesses were examined. The evidence showed that the addition produced a bolder and better risen loaf, and had a distinct bleaching effect. The baking process released an atom of oxygen, but there was no evidence that acid potassium sulphate remained when the flour was used. The protein content of the flour was good. The Stipendiary dismissed the prosecution, being of opinion: (1) That the potassium persulphate was introduced in the course of the preparation of the flour, as an article of commerce, in a fit state for consumption. (2) That the addition did not prejudice the purchaser, seeing that it actually improved the flour, was not put in to conceal inferior quality, and was introduced in such small quantities as to be wholly innocuous in its effects. He allowed no costs, and suggested that the millers would be well advised, if they again used persulphate, to declare its presence (*Grocer*, 1913, Aug. 2, Sept. 20, Dec. 6; 1914, Feb. 7; *B.F.J.*, 1913, 155, 175; 1914, 10, 51).

Birmingham. Maize flour 7%. The bag was marked "The new ideal biscuit flour" and also "Guaranteed genuine." A master baker stated that biscuit flour was the highest quality of wheat flour, and that he had never heard of maize flour, which was about 25% cheaper than wheat flour, being used. Fine £5, which, it was stated, the wholesaler would pay (*Grocer*, 1915, Aug. 14).

Sutton Coldfield. Maize meal 3 %. The fine and costs amounted to £4 7s. (*Grocer*, 1915, Sept. 4 ; *B.F.J.*, 1915, 195).

Omagh. Acid calcium phosphate 2.75 %. Evidence was given that the addition was made by millers to improve the flour, and that it might be more easily baked. The magistrates dismissed the case, being of opinion that the addition was not deleterious (*Grocer*, 1926, Nov. 20 ; *Analyst*, 1927, 52, 30).

PROSECUTION FOR DIABETIC FLOUR. *Salford.* Not diabetic flour, but consisted entirely of wheat flour. The price paid was 1s. 3d. for 3 lb., while the retail price of flour at that date was 2d. per lb. The Stipendiary fined the defendant £5, and said he must not sell it as diabetic flour again. A summons against the millers was dismissed as the retailer had signed a delivery note describing it as "special whole flour" (*Analyst*, 1930, 55, 41 ; *B.F.J.*, 1929, 128).

SELF-RAISING FLOUR

The Bread Acts Amendment Act, 1922, gave permission to add to flour ingredients suitable for the making of cakes or puddings. Power was also given to the Minister of Health to make Regulations in relation to such ingredients.

A report by Hamill to the L.G.B. in 1911 (Food Report, No. 13) stated that the usual composition of self-raising flour was about 6 lb. of acid calcium phosphate and 3 lb. of sodium bicarbonate to a sack of 280 lb. flour. In a small proportion of makes, tartaric acid or cream of tartar takes the place of the acid phosphate. The addition of self-raising ingredients may increase the "strength," and enable an inferior flour to be used.

A large proportion of the Birmingham samples examined from 1922-8 were phosphatic ; the range of ash was as follows :—

ASH IN PHOSPHATIC SELF-RAISING FLOUR, 1922-8 (Ninety-six Samples)

Percentage of ash	1.08-	1.5-	2.0-	2.5-	3.0-3.56	Total
Percentage of samples	12	47	20	16	5	100

In the next table the range of P_2O_5 in phosphatic flours is given, and also the corresponding percentage of $CaH_4P_2O_8$, on the assumption that no tri-calcic phosphate ($Ca_3P_2O_8$) is present. The figures are the maximum possible, as more or less tri-calcic phosphate is present in the commercial acid phosphate.

PHOSPHATE IN SELF-RAISING FLOUR (Eighty-one Samples)

Percentage of P_2O_5	0.24-	0.5-	1.0-	1.5-1.7	Total
Equivalent percentage of $CaH_4P_2O_8$	0.4-	0.8-	1.4-	2.5-2.8	
Percentage of samples	4	58	34	4	100

As $\text{CaH}_4\text{P}_2\text{O}_8$ is neutral to methyl orange ; by the use of this indicator sodium bicarbonate can be titrated in its presence, but two-thirds of any tri-calcic phosphate will also be neutralised, and so the following results will be rather too high :—

SODIUM BICARBONATE IN SELF-RAISING FLOUR (Eighty-one Samples)

Percentage of NaHCO_3	. . .	0.8—	1.0—	1.5—	2.0–2.7	Total
Equivalent percentage of CO_2	. . .	0.4—	0.5—	0.8—	1.0–1.4	
Percentage of samples	. . .	7	31	41	21	100

Although the figures in the two previous tables are too high, they will give an indication of the usual range of composition. With phosphate powders, the alkalinity calculated to Na_2O added to the phosphate calculated to CaP_2O_6 should be approximately equal to the amount of ash. With a tartrate sample, the alkalinity will not equal the total NaHCO_3 , but only the excess over the tartaric acid present. In this case the ash will be greater than the sum of the two constituents mentioned, and the presence of CaSO_4 will have a similar effect.

In 1929–31, the ash was determined in forty-eight samples ; 19 % of them contained 1.2–, 39 % contained 1.5–, and 42 % 2.0–2.8 % of ash.

The most important figure for self-raising flour is the amount of CO_2 set free on adding water. Twenty-eight samples examined in 1927–30 yielded 0.10 % to 0.50 %, and twenty-five of them gave 0.15–0.36 % ; probably some of the samples had lost strength by keeping. Though the value of the flour depends on this figure, I know of no attempt to fix a standard.

The amount of CO_2 yielded by acid is higher than that yielded by water, as excess of NaHCO_3 is used, and some of it is probably given off during baking (cp. *Macara, S.P.A.*, 1915, **40**, 272). Eleven samples varied from 0.35 % of total CO_2 to 0.51 %, the average being 0.42 %.

Calcium acid phosphate prepared by treating bone ash with sulphuric acid may contain as much as 50 % of calcium sulphate. Cripps (Bournemouth Report, 1908) found from 11 % to 33 %. His experiments showed that during baking 30–60 % of the calcium sulphate was changed into sodium sulphate and calcium carbonate. Hamill (*op. cit.*) stated that sometimes calcium sulphate had been deliberately added to acid phosphate. He stated that by using phosphoric acid in place of sulphuric acid there was no difficulty in preparing an article containing less than 5 % of calcium sulphate, and recommended that 10 % should be taken as the maximum limit. In a commercial acid phosphate containing 10 % of calcium sulphate, the percentage of P_2O_5 would be eight times that of the

SO_3 , but he recommended using the ratio of 7 to 1, so as to give the manufacturer the advantage of the margin of error.

The amount of SO_3 was determined in thirty-eight samples during 1911-27, and in thirty-six of them the percentage of SO_3 did not exceed one-seventh of the percentage of P_2O_5 ; another sample had an excess of only 0.06 %. These figures indicate that the one-seventh ratio is not too severe.

If impure sulphuric acid be used in preparing the superphosphate, arsenic may be introduced. One hundred and sixteen samples examined during 1916-31 were tested for arsenic, but the impurity was absent or less than 1 part per million. Lead is another possible impurity; twenty-three of the twenty-five samples examined in 1931 contained 0.4 parts per million, the others had 5 and 6 parts respectively. Of the sixty-three samples examined 1927-31, 11 % contained persulphate or peroxide.

Below are given comparative figures for water extract :—

WATER EXTRACT FROM FLOURS

	Plain Flour.	Self-raising Flour.	
		Tartrate.	Phosphate.
Percentage of organic extract	10.2	8.3	6.1
Percentage of ash	0.4	1.6	1.5
Percentage total water extract	10.6	9.9	7.6
No. of samples	5	10	32

The figures show that the self-raising ingredients diminish the soluble organic matter, particularly when phosphate is used.

Lerrigo's method (*S.P.A.*, 1926, 51, 180) detects 0.1 % of tartaric acid :—Shake about 4 gm. thoroughly with 20 ml. water, and filter after two minutes. Add a few drops of 1.5 E.AmHO to 10 ml. filtrate and shake. Add about 50 mgm. AgNO_3 in powder, put the test tube in water at 70°C .; do not shake till after two minutes. The turbid liquid produced with flour is pale yellow, but in the presence of tartrate light grey to almost black.

In 1908, five Birmingham samples containing 8-40 % of maize flour were condemned. A deputation from the Grocers' Association asked the Health Committee for permission to use 5 % maize flour, as a little would not matter, though maize was less nutritious and cheaper than wheat. Apparently the chemicals were first mixed with a small proportion of flour, and maize was thought to be drier than wheat and therefore less liable to cause decomposition. Subsequently a deputation from the Master Bakers' Association formed the Committee that the use of maize was unnecessary and plenty of self-raising flour free from it was sold. A local maker of large quantities of self-raising flour wrote and stated that 20-40 % of maize was being added for extra profit, and that if maize was used nothing less than 5 % was necessary (*B.F.J.*, 1899, 40; *F. & S.*, 1899, Jan. 21, Feb. 4, March 18).

In only one of the 350 samples examined in 1898-1930 was there an excess of calcium sulphate. That sample yielded 2.4 % of ash, 0.71 % of P_2O_5 , and 0.42 % of SO_3 . Subtraction of one-seventh of the P_2O_5 (0.10 %) from the SO_3 gave 0.32 % of excess; this multiplied by 1.7 gave 0.54 % of calcium sulphate in excess of the limit of 10 %. On one occasion plain flour was sold as self-raising flour.

Of the samples of self-raising flour examined in England and Wales, 1919-30, 1.0 % were adulterated.

ANALYSIS. The same methods may be used as for flour, with the following additions :—

Carbon Dioxide. Using 5 gm. determine the amount of CO_2 liberated by water and by acid, by Hepburn's method.

Alkalinity. Boil 5 gm. with 5 ml. N/2 HCl and about 100 ml. of water for a few minutes, or if a tartrate powder, long enough to boil off all the CO_2 . For tartrate powders use phenol phthalein as indicator and methyl orange for phosphate powders, cool and titrate back with N/2 NaHO.

PROSECUTIONS FOR SELF-RAISING FLOUR. *Southampton.* Alum, or some equivalent preparation, 50 grains per lb., and calculated to be injurious to health. The Public Analyst did not say that alum was present as such, but, in his opinion, the preparation had the same effect on the stomach as alum itself. The summons was dismissed, as the analyst admitted that there was no alum in the flour (*F. & S.*, 1895, July 27).

Liverpool. Alum upwards of 25 grains per lb. For the defence, it was pleaded that the stuff was a chemically prepared composition, and therefore there was no case. Prosecution dismissed (*F. & S.*, 1896, Aug. 22).

Wednesbury. Two samples, bought from a manufacturer, each contained 20 % of maize. The Public Analyst stated that the adulteration was detrimental to bread making, and that there was a difference of about 2 % between the nutritive values of wheat and maize. It had been claimed that maize flour was drier than wheat flour, and therefore more suitable as a "buffer" to prevent action during the mixing of the ingredients. He had, however, found the quantity of water to be about the same, and a dry wheat flour could be used. The proportion of maize flour, if used, need not exceed 3 %. The manufacturer was ordered to pay £10 14s. in fines and costs, and two of his customers were fined £1 and costs each (*F. & S.*, 1898, Nov. 19).

Luton. Sulphate of lime 58 grains per lb., or more than four times as much as was regarded as unavoidable. The Medical Officer of Health said that when people regularly ate food containing calcium sulphate indigestion and ill-health followed. It had the effect of an irritant poison, and he considered it injurious to health,

particularly in the case of young people. Fines and costs 83s. (*Grocer*, 1911, Nov. 11; *B.F.J.*, 1911, 219).

Wolverhampton. Sulphate of lime 1.34 %, being an excess over 10 %, in the 2.53 % of the acid phosphate used, which contained 57 % of sulphate of lime. Evidence was given that the article was made from 228 lb. flour, 7 lb. of "tar-tar-cream," and 3½ lb. of carbonate of soda. The manufacturer paid £8 10s. in fines and costs, and two of his customers paid 2s. 6d. each (*B.F.J.*, 1913, 40).

Bournemouth. Acid phosphate 0.58 %, which contained 22 % of calcium sulphate, whereas it should not have contained more than 10 %. For the defence, it was claimed that the Inspector got what he asked for, and was not prejudiced, as there was no standard for self-raising flour. Case dismissed (*Grocer*, 1916, Nov.).

London, Old Street. Flour which was not self-raising. Stated to be due to plain flour being put in the wrong bag. Fine £1 (*B.F.J.*, 1917, 17).

London, Lambeth. Flour which was not self-raising, sold by two retailers, had been supplied by the same wholesaler. Imperfect mixing was said to have resulted from two blades of the mixing machine having been broken. Dismissed on payment of costs (*Grocer*, 1919, March 8; *B.F.J.*, 1919, 40).

Wimbledon. Calcium sulphate 96 grains per lb. Fine £20 (*B.F.J.*, 1920, 29).

PUDDING POWDER, ETC.

Of the samples of "pudding powder" analysed in England and Wales, 1920-30, 1.9 % were adulterated, and only one of the 513 samples of "blanc mange powder" examined during the same period.

PROSECUTIONS FOR PUDDING POWDER. *St. Helens*. Sulphate of lime 15.3 %. Fine £10 (*Grocer*, 1917, July 14).

Brentford. Calcium sulphate 38.8 %, 36.6 %, 41.4 %, with $\frac{1}{13}$ grain arsenic per lb. Fine £2 in each case (*B.F.J.*, 1917, 161).

SEMOLINA, MACARONI, VERMICELLI

Semolina is prepared from wheat which contains a large proportion of gluten, as that grown in Manitoba and Algiers. It is derived from the less starchy part of the grain (Balland, *Analyst*, 1898, 23, 178). Hamill (Food Report, No. 14, 1911, p. 43) states that it is merely a trade name for the coarser fragments of endosperm produced in the break-roller system, and is incapable of exact description.

Macaroni and vermicelli are made in Italy, France, and to some extent in other countries, from semolina. The dough made from the flour is placed in a metal cylinder with a perforated plate at one

end and a plunger at the other, and pressure applied. The tubes or sticks when dried are ready for the market (Winton and Ogden, Connecticut Report, *B.F.J.*, 1903, 35). Turmeric and nitro-colours are extracted by long-continued shaking with alcohol, and orange coal-tar dyes by shaking with a mixture of 10 of alcohol and 1 of HCl (Winton and Ogden). Analyses of the articles are given by Balland (*op. cit.*).

ASH OF SEMOLINA (Fifty-six Samples)

Percentage of ash . . .	0.2—	0.4—	0.6—	0.7–0.84	Total
Percentage of samples . .	7	22	48	23	100

It may be pointed out that nearly half the samples were between 0.6–0.7 %. Of macaroni, sixteen samples were examined ; the range of ash was 0.3–0.8 %, and that of moisture 10.1–12.9 %. Some of the samples were examined in greater detail :—

AVERAGE COMPOSITION OF BIRMINGHAM SAMPLES

	Samples.	Protein.	Fat.	Ash.	Moisture.
Semolina . . .	10	11.0	1.7	0.67	12.2
Macaroni . . .	10	11.6	1.0	0.51	11.9
Vermicelli . . .	4	11.8	0.5	0.62	12.0
Egg Vermicelli .	1	13.1	0.5	0.72	10.7

The label on the sample of “ Egg Vermicelli ” claimed that with 4 oz. of it only one egg need be used, instead of two eggs with ordinary vermicelli. The vendor was cautioned for the false label, as 100 grains of fat and about 300 grains of protein should have been present in 4 oz. if the label had been correct, but only 9 grains of fat and 230 grains of protein were actually present. The article had been artificially coloured with coal-tar dye, probably to make it look more egg-like.

According to the Ministry of Health Reports, 1,210 samples of “ wheat preparations ” were examined in England and Wales, 1920–30, and only seven of them were reported to be adulterated.

RICE

In 1905, Matthes and Müller (*Analyst*, 1905, **30**, 206) drew attention to the coating of rice with talc, seven out of fifteen samples containing 0.24–1.0 %. In the next year Cribb and Richards (*Analyst*, 1906, **31**, 40) reported that eleven dull official samples of rice gave 0.29–0.57 % of ash, while seventeen polished samples had 0.52–2.22 % of ash. The variation in the proportion of total ash was chiefly due to the insoluble part, as that soluble in acid only varied from 0.28 % to 0.57 %. The coating matter, under the microscope, appeared as transparent angular particles of irregular outline. It contained about 60 % of silica (SiO_2) and about 24 % of magnesia (MgO), and was similar in composition to French chalk, steatite and talc.

Hamill made a report to the L.G.B. : " On ' facing ' and other methods of preparing rice for sale " (No. 8 Report of Inspectors of Food, 1909). He was informed that during milling rice was polished, but talc, if used, hardly affected the ash. The subsequent glazing was done with talc mixed with glucose, glycerin and starch paste, and added about 0.2 % of mineral matter. In some cases, instead of glazing, the grains were made translucent by oiling with mineral oil. Any improvement in appearance is lost on cooking.

Hamill considered that the presence of 1 % or 2 % of insoluble and possibly irritating matter might in some circumstances be definitely prejudicial to health, especially as this food is often given in considerable quantities to children and invalids. He quotes a United States Law that talc may only be used if its presence is declared on the label, with directions for removing it. Lowe and Taylor (*Analyst*, 1910, **35**, 479) described a pancreatic calculus containing much silica and alumina. It was attributed to the patient consuming a rice pudding every day for months, made of rice coated with 0.9 % of facing. Issoglio (*Analyst*, 1920, **45**, 451) has compared the detailed composition of seven qualities of Italian rice ; they show that polishing decreases the valuable nutritive elements in rice.

Rice brokers have claimed that talc acts as a preservative against the attack of weevils and other insects ; but Hamill could find no positive evidence in support of this, and quoted Hefelmann, who stated that even 1.6 % of steatite did not protect rice from weevils.

In 1909, five of the twelve samples of rice examined in Birmingham were faced with 0.61 % to 0.88 % of mineral matter, and the attention of the local Grocers' Association was called to the matter. From 1912 to 1922, five of the 715 samples were similarly faced, but none of those examined in subsequent years.

ASH IN RICE (1,252 Samples) AND GROUND RICE (Ninety-two Samples)

Total percentage of ash	RICE.			GROUND RICE.	
	1909-14	1915-22	1923-31	1913-21	1924-31
Less than 0.3 . . .	2	12	17	0	25
0.3-	23	50	75	52	61
0.5-	42	24	6	44	14
0.7-	33	14	2	4	0
	<hr/>	<hr/>	<hr/>	<hr/>	<hr/>
Maximum	100	100	100	100	100
	1.32	0.97	0.95	0.72	0.58

Comparison of the figures for the three periods indicates a great diminution in the amount of ash. In the first period only 25 % of the samples of rice contained less than 0.5 % of ash, while in the last period 90 % were below this figure. Ground rice, all samples of which were passed as genuine, also showed an improvement.

Krzizan (*Analyst*, 1906, **31**, 263) gave a method for determining the facing by removing it by treatment with hydrogen peroxide and ammonia, and treating it with chromic acid. The method is tedious, and the writer obtained the same results by allowing the ash of 10 gm. of rice to stand with 10 ml. cold E.HCl, followed by two treatments with 10 ml. of water. The presence of talc makes the ash infusible.

INSOLUBLE ASH IN RICE, 1909-14 (138 Samples)

Percentages of insoluble ash	0-	0.3-	0.6-0.88	Total
Percentage of samples	51	43	6	100

The average composition of seven samples of rice examined in 1917 was as follows: Nitrogen 1.07 % (= 6.8 % protein), fat 0.61 %, ash 0.56 %, moisture 11.8 %, fibre 0.28 %.

Official reports indicate that 13.2 % of the samples of rice analysed in England and Wales, 1905-13, were adulterated, and 3.7 % of those examined 1920-30.

PROSECUTIONS FOR GROUND RICE. *Wood Green.* Maize starch 15 %. Fine 5s. and costs (*B.F.J.*, 1904, 93).

Bromley. Entirely a preparation of wheat. The defence was that more expensive semolina had been sold in error. Case dismissed (*Grocer*, 1920, July 31).

PROSECUTIONS FOR RICE. *Ruthin.* Steatite 1.01 %. The packet was marked "Choicest growth." The Medical Officer of Health considered that rice coated to any large extent with steatite would be injurious to health, as it would retard digestion. Fine £5 and costs (*Grocer*, 1911, July 1; *B.F.J.*, 1911, 138).

Gainsborough. Extraneous mineral matter 0.54 %, or 0.04 % in excess of the limit. Subsequently the Government analysts reported 0.57 %. Fine £2 and costs. Notice of appeal was given, but no further action was taken (*B.F.J.*, 1912, 198; *Grocer*, 1912, Oct. 19; 1913, Jan. 18).

Blyth. Talc or French chalk 0.8 %. An analyst for the defence said talc would not affect infants or invalids, especially in such a small percentage. Talc cost 10s. per cwt. and rice 12s. Case dismissed (*Grocer*, 1913, July 5).

Moneymore. Extraneous mineral matter 1.1 %. Case dismissed (*Grocer*, 1928, Aug. 18).

Derry. Extraneous mineral matter or glazing material 0.86 %. The Government analysts subsequently found 0.69 %. Fine and costs £3 2s. 6d. (*B.F.J.*, 1928, 119).

Tynan. Extraneous mineral matter 0.8 %, only 0.5 % being allowed. The sample was stated to have been taken from the bottom of the sack, and possibly dust and dirt had got into it. Costs paid (*Grocer*, 1930, April 12).

PEARL BARLEY

Good samples of pearl barley are uniformly white as the result of milling, a process which Church ("Foods") states may result in 60 % of waste. If the barley is not uniform in size, or the milling is less thoroughly done, some of the grains will not have the brown coats removed, and the appearance of the sample will be spoiled. The appearance of such samples was found by experiment to be much improved by facing with talc or rice flour, stuck on with glucose and glycerin. The average weight of unfaced grains was 17 mg. and of faced ones 20 mg.

Pearl barley is liable to the attack of grain weevils, which are small beetles about one-eighth of an inch long. These insects make grains partially or entirely black by their attack. Millers assert that facing protects the grains, but Liverseege and Hawley (*J.S.C.I.*, 1915, 203) found little difference between faced and unfaced samples in the proportion of grains attacked.

Matthes and Müller found talc up to 0.63 % in one-third of the samples they examined (*Analyst*, 1905, **30**, 206). Hamill, in his report on rice (No. 8 Report of Inspectors of Food, 1909), mentions the extension of facing to pearl barley. The *B.F.J.* records a case of a regular consumer of pearl barley suffering from severe dyspepsia, after taking an article faced with French chalk (1906, 221).

In 1913-4, one-third of the Birmingham samples were faced, either with rice, up to 2 % being present, or with talc, or with both. Of the eighty-six samples examined 1910-4, 9 % had 0.7-0.85 % of ash insoluble in HCl, 8 % had 0.3-0.6 %, 9 % had 0.1-0.2 %, and 74 % contained less than 0.1 % of ash insoluble in acid. Two vendors were prosecuted, and a number, including some wholesale dealers, cautioned, and that form of adulteration ceased. The following table shows the range of ash in two periods :—

ASH IN PEARL BARLEY

Percentage of ash	0.62-	0.8-	1.0-	1.2-1.68	Total
Percentage of samples :					
1912-20	9	39	35	17	100
1921-30	19	53	24	4	100

Comparison of the two periods shows that there has been a marked fall in the proportion of samples containing high ash, and therefore faced.

Some samples of pearl barley are free from dust ; in other cases, if the dust present be put in water, small brown particles may be seen by the naked eye, and the majority of these on examination with the microscope are seen to be mites, either alive or dead. Mites have been found in faced samples. The diminution in the use

of facing indicated above has been accompanied by an increase in the cleanness of pearl barley. In the six years 1918–23, 55 % of the samples contained mites, but in the next seven years the percentage fell to 27. Pearl barley should obviously be washed before use.

Samples of pearl barley were first tested for sulphur dioxide in 1927–8, and quantities up to 120 parts per million were found. Retail and wholesale vendors were cautioned, and in 1929 that impurity was not detected. Probably bleaching had for some time taken the place of facing.

Of the samples of “barley” examined in England and Wales in 1904–5, 31 % were adulterated with mineral facing. During 1906–13, the proportion of adulteration was 4.1 %, and during 1920–30, 3.1 %.

ANALYSIS. The facing was determined in the same way as on rice, and in some case the talc was removed by washing 10 gm. and determining the ash of the washed grains and the washings separately. In one case the washings accounted for 0.9 % of total ash, and 0.68 % of insoluble ash, and the washed grains for only 0.69 % and 0.08 %, respectively.

The washings of rice faced samples were examined by the microscope and the proportions of rice and barley determined, then the solid matter in the washings was determined; the soluble matter in the washings, glucose, etc., amounted to 0.5–1.0 %.

PROSECUTIONS. *Peterborough.* Rice 12.5 %. The defendant stated that he had served chicken food by mistake; he was ordered to pay 7s. 6d. (*B.F.J.*, 1913, 230).

Kingston. French chalk 0.5 %. It was stated that a person drinking barley water made from such a sample might consume $\frac{1}{4}$ to $\frac{1}{2}$ drachm of French chalk per day. The defendant's warranty proved unavailing, as he had continued to sell it after he had been warned by the County Council. He paid 64s. as fine and costs (*B.F.J.*, 1914, 158).

Birmingham. Rice facing 2 %, to improve the appearance of the article. The defendant was ordered to pay 4s. costs only, on undertaking not to sell pearl barley without a guarantee of genuineness (*Grocer*, 1914, Oct. 31).

London, Old Street. Sulphur dioxide 180 parts per million. Ordered to pay 21s. costs (*Grocer*, 1928, June 9; *B.F.J.*, 1928, 70).

London, Old Street. Sulphur dioxide 50 parts per million. Defendant's analyst found 55 parts. Fine £2 (*Grocer*, 1928, June 16, 30, July 7).

OATMEAL, ROLLED OATS

Church (“Foods”) states that before milling it is necessary to dry the oats in a kiln to facilitate the removal of the husk, which otherwise adheres firmly to the oat grain.

Analyses of oat preparations have been given by Dyer (*S.P.A.*, 1901, **26**, 153) and by Maclaurin (*Analyst*, 1924, **49**, 184).

In 1877 a paper by Cleaver (*S.P.A.*, **1**, 187) initiated a discussion at the Society of Public Analysts on the presence of barley meal in oatmeal. The opinion was expressed that anything more than 1 % or at most 2 %, was adulteration. White (*S.P.A.*, 1895, **20**, 30) has directed attention to the adulteration of oatmeal with maize, of which he found up to 3 %.

Adulteration of rolled oats is improbable; possibly another flattened grain might be substituted: 178 Birmingham samples were genuine.

COMPOSITION OF OATMEAL AND ROLLED OATS, 1916-7 (Six Samples each)

Percentages.	Moisture.	Fat.	Protein.	Ash.
Oatmeal . .	8.6	7.3	13.9	1.8
Rolled Oats .	7.7	6.1	14.4	1.8

Oatmeal is not easy to ash, and for some years the sulphated ash was determined—shown by the following table to be higher than the ash. Sulphation was discontinued as being of little advantage.

ASH IN OATMEAL AND ROLLED OATS, SULPHATED ASH

Percentages.	Rolled Oats. Ash. 1920-31.	Oatmeal. Ash. 1900-18.	Oatmeal. Ash. 1919-31.	Oatmeal. Sulphated Ash. 1895-9.
1.1- . . .	6	2	6	2
1.5- . . .	55	23	33	4
1.7- . . .	39	63	55	42
2.0- . . .	0	10	6	40
2.4- . . .	0	2	0	8
2.7-2.9 . .	0	0	0	4
	<hr/>	<hr/>	<hr/>	<hr/>
	100	100	100	100
Samples examined	192	239	445	48

Owing to the fusion of the ash, the amount of it insoluble in acid may be overestimated if treatment is insufficient. Two of the seventy samples in which it was determined gave 0.12 % and 0.16 %; the others varied from 0.01-0.09 %. The average of all the samples was 0.04 %.

In 1913-4, samples of oatmeal yielded 2.5 % of ash, and 0.4-0.7 % ash insoluble in acid. This high result was due to the presence of husk. The crude fibre amounted to about 5 %, while samples giving no evidence of husk had 0.4-2.0 %. The husk was best determined by rubbing 10 gm. of the oatmeal with water in a mortar, and washing away the starchy matter by levigation. The

residual husk was dried and weighed. Four adulterated samples thus treated yielded 4.0–8.3 % of husk, while the amount in fifty genuine samples did not exceed 0.12 %.

In 1876–7, no fewer than six of the fifteen samples of oatmeal examined in Birmingham were adulterated with 10–30 % of barley meal. From 1883–1931, 1,417 samples of oatmeal were examined; of them 0.8 % were adulterated with barley meal and 0.4 % with husk.

In 1899 the L.G.B. Report mentioned a sample of oatmeal which was so much adulterated with refuse or sweepings as to be more fit for pig food than human porridge. In 1894 no less than 7.2 % of the samples examined in England and Wales were adulterated; the next year the proportion fell to 2.5 %; in 1908, 1.9 % were condemned. In subsequent years rarely more than 1 % of the samples were adulterated, the average being about 0.5 %.

PROSECUTIONS. *West Bromwich.* Meal, chiefly barley, 24 %. The Government analysts found 22 % of meal, not oat, in the sample. Fine 10s. (*Analyst*, 1878, 2, 84).

Barnard Castle. Barley meal 16 %. Vendor stated that barley and oats were grown together, and that mixing was unavoidable. He was ordered to pay costs (*F. & S.*, 1893, May 20).

Heanor. Maize 3 %. The article was sold by a miller and two of his customers. He maintained that it was sold as a cattle food, and that the presence of the maize was accidental, or due to its use as an aid to grinding. He was fined £1, and his customers were ordered to pay costs only (*F. & S.*, 1894, Dec. 15).

Measham. A substance not oatmeal 10 %. The vendor pleaded guilty and was ordered to pay costs (*B.F.J.*, 1900, 204).

CORNFLOUR

In 1856, a process of treating maize was patented, and since then the product has been sold as cornflour. Much money has been spent on prosecutions owing to some manufacturers claiming to sell a rice preparation as cornflour. In one appeal, after a long hearing, the chairman was satisfied that cornflour was the starch of Indian corn only. In a later case a magistrate dismissed a summons, holding that rice was just as good as maize, and that the purchaser was not prejudiced. Although the difference in chemical composition is trivial, both being nearly pure starch, the writer considers that a purchaser is prejudiced if he is supplied with a different article from the one demanded. According to evidence given by a lecturer in cooking, who had made experiments, rice has decidedly less stiffening power than maize. There is also the possibility that the vitamin content of the two substances may be

different. In some cases the substitute has been called "British" cornflour, as if rice was more British than maize.

Stubbs (*S.P.A.*, 1926, **51**, 400) has stated that 133 out of 139 samples of cornflour were entirely maize, and has described sweet potato starch which he found in other samples. The twenty-five Birmingham samples were maize, and the experience of other analysts has been similar. The packets containing rice cornflour are now marked "Made from Rice" (cp. *Analyst*, 1927, **52**, 75). There is the possibility of a retailer opening a packet, selling part of the contents, and neglecting to give proper intimation to the purchaser.

Two varieties of cornflour are on the market. Of the samples analysed in Birmingham, 1917-28, 52 % yielded less than 0.2 % of ash, while 40 % yielded 0.4-0.6 %. The ashes of the former were almost neutral to methyl orange, but the alkalinity of the latter varied from 3-4 N.v/w. Cornflour itself is usually slightly acid (2.5 N.v/w) to phenol phthalein, but one sample was alkaline.

COMPOSITION OF WAR-TIME SAMPLES, 1917

	Protein.	Fat.	Ash.	Moisture.
Cornflour . .	0.3	0.2	0.3	11.8
Flaked Maize .	3.6	1.5	0.5	11.7
Maize Meal . .	8.9	2.8	0.8	9.6

Drake-Law (*Analyst*, 1927, **52**, 353) found 100 parts of sulphur dioxide in an American cornflour, and 30 parts in a Dutch one. A British sample was found to be free from sulphur dioxide, but after standing four weeks it had 25 parts per million.

During 1905-13, 1.3 % of the samples of cornflour examined in England and Wales were adulterated, and 0.9 % of those during 1920-30.

PROSECUTIONS FOR CORNFLOUR. *Kensington.* Starch having the characters of rice starch 100 %. The article was a Belgian preparation labelled "The new cornflour. . . Far superior to any other." The magistrates convicted the defendant, who appealed to the Clerkenwell Sessions. The Public Analyst then stated that cornflour was a preparation of maize, and not of rice, and that the flavour of the two preparations was different. The article in question had 1.2 % of mineral matter and was powerfully alkaline. Other public analysts gave corroborative evidence, and trade witnesses stated that they considered cornflour to be made from maize. Evidence was also given that cornflour had been made from maize by a special process since 1856. A lecturer on cookery stated that half as much again of the article was required as of cornflour to make a stiff blancmange. The chairman stated that he was satisfied that, both in England and America, cornflour was only the starch of Indian corn. The appeal was dismissed, the fine

reduced to £1 without costs, and the defendant undertook to mark each packet "Prepared from rice" (*B.F.J.*, 1902, 221, 257, 269).

Loftus. Principally potato starch. The wholesale dealers stated that, by a mistake, more expensive "farina" had been packed instead of cornflour. They undertook to pay the defendant's costs (9s.) and expenses (*Grocer*, 1907, Feb. 2; *B.F.J.*, 1907, 29).

Kensington. Wheat starch 100 %. Fine and costs 41s. (*Grocer*, 1918, Nov. 23; *B.F.J.*, 1918, 131).

London, South-Western. Potato flour 10 %. It was marked "Pure cornflour." Fine £2 and costs (*Grocer*, 1919, July 2; *B.F.J.*, 1919, 75).

London, South-Western. Tapioca flour 90 %. The magistrate was satisfied that the defendant had acted in good faith, and ordered him to pay one guinea costs only (*Grocer*, 1919, July 2; *B.F.J.*, 1919, 75).

London, Lambeth. Rice starch 100 %. The Public Analyst admitted that there was no difference between rice starch and maize starch from the food point of view, and that he had not tried if there was any difference in taste or flavour. Analytical and other evidence was given that cornflour was maize starch. The magistrate dismissed the case and allowed 25 guineas costs. He said the evidence did not justify him in holding that those who sold maize cornflour had any right to appropriate to themselves exclusively the right to the name, and also, that the evidence did not show any prejudice to the purchaser (*Grocer*, 1919, Aug. 2, 16).

Wigan. Sweet potato flour. It was stated that the article had been made by the Government in 1918, when maize had been difficult to get, and that the packet had been overlooked. The chairman dismissed the case on payment of costs, the article having been withdrawn from sale (*Grocer*, 1921, April 6).

PROSECUTIONS FOR INDIAN MEAL. *Newtown Hamilton*. Deficient in oil natural to Indian meal to the extent of at least 62 %. At least 4 % of oil should be present, and sometimes there was 6 %. For the defence it was stated that human food meal will not carry more than 1.52 % of oil, and that if more were present it would not keep and would have a bad colour. In the process of manufacture it was necessary to remove the germ, which contained nearly all the oil, and the bran, which contained 1 %. The vendor was fined 1d. and 2s. costs. On appeal to the Dublin King's Bench Division, the justices quashed the conviction, as the Inspector was not deceived, having received Indian meal specially prepared for human food, and known to the public as such (*B.F.J.*, 1916, 275, 311).

SAGO

Eighteen samples of sago bought in Birmingham in 1888 and 1897 were genuine, but each of the ten samples bought in 1920-2

were tapioca. Tapioca and sago are similar in composition, but prepared from very different plants, and the writer feels strongly that the substitution of one article for the other is to the prejudice of the purchaser.

Seed tapioca is slightly whiter than sago—whiteness which may be due to bleaching. It is possible that a purchaser, who knew that the root from which tapioca is prepared contains prussic acid, might prefer sago, which is not so accompanied.

There is not much difference in the relative cost of the two articles, and apparently sometimes sago is the dearer, and at others tapioca. One cannot agree that a substitution that is undeclared, undiscovered, and unpunished, becomes legitimate merely through lapse of time. If the position is as some vendors maintain and the substitution were notified by a label, such as: "Pearl Tapioca, sometimes sold as Sago," there would be no prejudice, and members of the public would know what they were buying, and would soon decide which article was preferred. In some districts there is little demand for genuine sago (see *Grocer*, 1931, May 9, June 13, July 18).

The practice of substitution has been strengthened by the assumption that the King's Bench appeal case, *Sandys v. Rhodes* (1903), settled a question of *fact* and not merely one of law. Owing to the illness of the Public Analyst, the original prosecution was dismissed by the magistrates without any expert evidence being given for the prosecution. Consequently the case stated for the opinion of the High Court was one-sided, and all the appeal settled was that the magistrates might decide that such a sale was not to the prejudice of the purchaser. Had the appeal been to Quarter Sessions on the question of fact, one feels that the result might have been different. It is notable that in a later prosecution a magistrate fined a defendant for the same offence, in spite of the appeal case being quoted.

Of the samples of sago examined in England and Wales, 1903–30, 10.2 % were reported adulterated.

PROSECUTIONS. *Andover.* Starch other than sago starch 50 %. The Public Analyst stated that owing to the heat used he was unable to say if any sago was present. For the defence it was stated that when "small sago" was asked for, pearl-seed tapioca was sold, which cost about 3s. per cwt. more. The Bench decided that there had been no prejudice, and dismissed the case (*F. & S.*, 1895, Aug. 10).

London, Kensington. Granular starchy matter, having the characteristics possessed by wheat and barley starch 12 %. Fine 10s. (*F. & S.*, 1897, July 17).

Derby, Tapioca 100 %. For the defence it was contended that it was the custom of the trade to sell tapioca as sago, that there was

no appreciable difference in the trade values, and that for a number of years the public had preferred the whiter tapioca. The justices came to the conclusion that there was no prejudice, as the public had an idea that what should be called pearl sago was tapioca, and dismissed the case. On appeal, the High Court held, *Sandys v. Rhodes* (1903), that the justices might find that such a sale was not to the prejudice of the purchaser (*B.F.J.*, 1903, 132).

London, Battersea. Tapioca, made into granules of the same size and shape as real pearl sago, 100 %. Evidence was given that sago cost 2s. 6d. to 3s. more per cwt. than tapioca. The Public Analyst stated that there were fundamental differences in regard to the taste, flavour and digestibility. The Medical Officer of Health considered that if given to a convalescent, the two articles would not have the same dietetic effect. The magistrate was satisfied that the sale was to the prejudice of the purchaser, although the price was similar, and fined the defendant 5s. He added that tradesmen must understand the all-important fact that when a particular article was asked for it must be supplied (*B.F.J.*, 1904, 17).

Ilminster. Rice 1.7 %, barley, vegetable fibre and the remains of the cocoon of some insect, probably moth, the living larvæ of which were present. Fine 10s. (*Grocer*, 1921, June).

Cambridge. Seed pearl tapioca. The Public Analyst stated that sago was a very delicate starch and very easily cooked; while tapioca was a tougher article, and when cooked, even for a considerable time, presented lumps of thick jelly, and was not so easily digested. The vendor was ordered to pay £2 12s. costs (*Grocer*, 1921, Sept. 17; *B.F.J.*, 1921, 99).

TAPIOCA

An analysis by Balland (*Analyst*, 1898, 23, 178) gives its composition as—water 12.8 %, fat 0.2 %, ash 0.4 %, cellulose 0.08 %, the rest being starch, etc. Willmott's figures for cassava starch are—moisture 14.14 %, protein 0.18 %, fat 0.03 %, fibre 0.03 %, ash 0.19 %, the remainder being starch (*P.J.*, 1929, Aug. 17).

During 1916–31, 512 samples were bought in Birmingham, and all passed as genuine. The ranges of ash were as follows:—0 %–44 %, 0.1–52 %, 0.2 % 3%, 0.4–0.6 % 1 %. The high ashes of the last three, bought in 1918, were due to sand, the amount insoluble in acid being 0.32–0.4 %. Thirty-one samples were examined for sulphur dioxide in 1927–9, but none was detected.

More than 5,000 samples of tapioca were examined in England and Wales during 1905–30; the proportion of adulteration was only 0.2 %.

ARROWROOT

The question "What is arrowroot?" has given rise to much controversy. After consulting authorities, one considers that the position is fairly put by Squire ("Companion to B.P.") :—"The name arrowroot is properly applied only to the starch derived from *Maranta Arundinacea*, but it is often loosely applied to the starch derived from other plants when used as food for invalids." The B.P. Codex (1923) simply states: "Maranta starch (arrowroot) from *Maranta Arundinacea*."

The description of potato and maize starches as "British arrowroot" indicates a desire in some quarters to degrade the word till it merely becomes a synonym for starch. The underlying suggestion, that the substance is obtained from the same plant grown in another country, is quite incorrect. I have a list of the botanical names of eleven different plants, from as many different natural orders, to each of which the name "arrowroot" has been applied with a geographical prefix.

All starches are not identical in properties. Langworthy and Deuel (*Analyst*, 1922, **47**, 356) found that small starches were more readily digested, and that there was a direct relationship between the size of the grains and the amount digested. The starches were eaten raw. It appears reasonable that the word "arrowroot," when unqualified, should be used only for the original article, *Maranta arrowroot*, in the same way that "cornflour" should be a maize product.

The following analyses are by Willimott (*P.J.*, 1929, Aug. 17) :—

COMPOSITION OF ARROWROOT AND POTATO STARCH

Percentages.	Moisture.	Protein.	Fat.	Fibre.	Ash.	Starch.
St. Vincent Arrowroot .	15.15	0.10	0.02	0.03	0.25	84.45
Potato Starch .	16.20	0.12	0.03	0.03	0.25	83.37

According to J. Campbell (*Lancet*, 1929, Aug. 10), recent experiments at the Lister Institute indicate that root starches, such as arrowroot, are superior to cereal starches in containing a hitherto unknown constituent, which, if not a vitamin, has an allied action. He is of opinion that the substitutes for arrowroot, particularly those from maize and potato, do not contain the dietetic properties of genuine arrowroot.

The *Pharmaceutical Journal* stated (1929, Aug. 17) that the principal source of arrowroot is now the island of St. Vincent, and that arrowroot has not been imported from Bermuda for many years.

The experience of Birmingham supports the position here taken. During 1881–1931, 2,475 samples of arrowroot have been examined,

in only three of the thirty-eight different years was adulteration detected. In 1887 a sample was tous-les-mois starch, in 1902 a vendor was fined 10s. for the sale of maize starch powder, and in 1928 five samples were adulterated with one-half to two-thirds tapioca starch. It was stated by one importer that a consignment of "Genuine St. Vincent Arrowroot" sent directly from the docks to the retailer was tapioca starch. The adulteration of some of the Birmingham samples was probably accounted for by the tapioca being emptied into a receptacle containing genuine arrowroot. There is a curious similarity between the 1928 samples and a prosecution for 50 % of tapioca in 1877. It was then stated that there was no known case of adulterated arrowroot having been sent from St. Vincent.

There is little variation in the amount of ash in arrowroot, the range found in 568 samples being : 0.02 %– 7 %, 0.1 %– 73 %, 0.2 %– 16 %, 0.3 %– 3 %, 0.44–0.48 % 1 %. The higher ashes were due to the presence of sandy matter, 0.26–0.34 % of ash insoluble in acid being found. Each of the fifteen samples analysed in 1928 was free from sulphur dioxide.

There is a great range in the market value of arrowroot ; genuine Bermuda may cost four times as much as the St. Vincent variety. Probably the dietetic value bears little relation to the cost.

During the years 1886–1930 over 10,000 samples of arrowroot were examined in England and Wales—0.8 % of them were adulterated ; 1928 was the worst year, 3.9 % of adulteration.

PROSECUTIONS. *London, Clapham Park.* Tapioca 50 %. The defendant stated that the article was sold as it came from St. Vincent, and that there was no known case of adulterated arrowroot having been sent. The Government analysts subsequently reported that not less than 25 % of tapioca flour was present (*Analyst*, 1877, 1, 81).

Ashford. Tous-les-mois starch. Evidence was given that the substitute was harmless, of similar value, and obtained from the importers as St. Vincent arrowroot. The case was dismissed as there was no fraudulent intent (*F. & S.*, 1896, May 30).

West London. Borax 100 %. The magistrates described it as a case of gross negligence, and fined the defendant £5 (*F. & S.*, 1897, April 17).

Birmingham. Maize starch powder. The article was stated to be one-third the cost of arrowroot. Fine 10s. (*B.F.J.*, 1902, 204).

Wigan. Maize starch 90 %, potato starch 10 %. The defendant was ordered to pay costs (*Grocer*, 1919, May 13).

London, Lambeth. Tapioca starch. Fine £2 2s. (*Grocer*, 1923, Jan. 20).

London, Watford. Cornflour at least 90 %. Fine £1 and costs (*Grocer*, 1915, Jan. 17).

Wigan. Starch foreign to arrowroot 99 % (sweet potato starch). The vendors failed to identify the article with any particular invoice and were ordered to pay costs (*Grocer*, 1930, March 22 ; *B.F.J.*, 1930, 45).

Padiham. Sweet potato starch 40 %, cream of tartar 9 %. The defendant proved a warranty and the case was dismissed (*Grocer*, 1930, March 29 ; *B.F.J.*, 1930, 35).

CHAPTER XIII

SUGAR ANALYSIS

Solution and clarification. Polarisation. Inversion of sucrose. Reduction, Fehling solution. Fermentation. Calculations, corrections, mixtures, constants and factors.

AMONG special methods for the determination of sucrose may be mentioned—a Government Laboratory Report on its determination in bread, buns and fruit cakes, including an estimation of the amount of sugar in fruits (*Analyst*, 1917, **42**, 293), and a Report by the Milk Products Sub-Committee on its determination in condensed milk (*S.P.A.*, 1930, **55**, 111); also, a lengthy paper by Monier-Williams on the latter subject (*S.P.A.*, 1928, **53**, 569).

Hinton and Macara have studied in detail the conditions in which dextrose and lactose are reduced by iodine, and in which sucrose and lævulose are only slightly altered (*S.P.A.*, 1924, **49**, 2). Later on they made a similar examination of the chloramine-T method, which they preferred and applied to milk products, and compared it with the polarimetric method (*S.P.A.*, 1927, **52**, 668).

Sugars have been compared as to their sweetening powers by Paul, who includes saccharin (*Analyst*, 1922, **47**, 261), and Beister, Wood and Wahlin (*Analyst*, 1926, **51**, 255). Another estimate gives the sweetening power of honey as 75 % of that of sucrose; maltose syrup as 30 %, and corn syrup as 20 % of it (*Y.B.P.*, 1920, 345).

SOLUTION AND CLARIFICATION. As the standard temperature for polarisation is 20° C., the flasks used should be calibrated at that temperature, and solutions be diluted to the mark on the flask only when that temperature is attained.

As the standard concentration for the polarisation is 10 w/v, if possible when several sugars are present, a weight of the substance analysed should be taken to yield a solution of about that strength of total sugars (*S.P.A.*, 1928, **53**, 573). Correction, if necessary, can be made as shown below (p. 161). With sucrose itself, the specific rotation of which is unaffected by the concentration, the use of a 20 w/v solution gives a more accurate result.

With turbid solutions the inclusion of a little alumina cream will promote clarification by filtration. Colouring matter may often be precipitated by lead acetate, or lead subacetate, but the latter in excess may remove dextrose and lævulose. After filtration the lead may be removed by solid potassium sulphate, ammonium oxalate,

or sodium phosphate, without appreciable increase of volume. Animal charcoal is sometimes useful, but the first amount filtered should be rejected. Dark-coloured syrup solutions may be bleached by Heron's method. To 100 ml. add 5 ml. of chlorine solution, let stand a minute or two till decolourised, then add excess of solution of sulphur dioxide, twist to mix, dilute to mark, well shake and filter if necessary. The reagent is made by triturating 1 oz. of bleaching powder with 4 oz. water, and filtering. It should be kept in a cold dark place.

With nitrogenous substances such as milk, the use of zinc ferrocyanide or phosphotungstic acid, may be advisable (*S.P.A.*, 1927, **52**, 676; 1930, **55**, 113). If there be a notable volume of precipitate, an allowance for its volume must be made (cp. *S.P.A.*, 1930, **55**, 116) (cp. p. 218).

POLARISATION. For general analytical work a polarimeter graduated in angular degrees is preferable. Monier-Williams has described an arrangement for maintaining a constant temperature in a polarimeter (*S.P.A.*, 1928, **53**, 580). Readings should not be taken till the temperature of the water is constant, as shown by a thermometer; if the temperature is not exactly 20° C., a correction can be made, as is shown below (p. 161). The ends of the polarimeter tube must not be screwed on tightly, or the strain in the glass may produce optical distortion. The zero of the instrument should be periodically tested, or there may be a constant error in a series of readings.

A sucrose solution must not be alkaline; by polarising in N/10 NaOH, 9.7 % of sucrose was indicated instead of 10 %, but with N/20 the deficiency was only 0.1 %. Boiling a 10 % solution of sucrose in water, or in N/100 NaOH, did not alter its rotation. The presence of 10 v/v of purified methylated spirit in a 9.0 w/v solution of sucrose slightly increased the polarisation—9.1 w/v being indicated. The addition of subacetate of lead and acetic acid to a 10 w/v solution of sucrose did not affect its polarisation.

If a substance contain both sucrose and citric or tartaric acid, for polarisation it should be dissolved in dilute alkali of such a strength that most of the acid is at once neutralised, or inversion may proceed before and during polarisation.

The muta-rotation of lactose may be avoided by dissolving in hot water, adding ammonia when cool, and neutralising after standing (*S.P.A.*, 1930, **55**, 114).

Methods for calculation, with constants and factors for correction of various sugars, are given in a subsequent section (p. 160).

Formulae for the calculation of the specific rotations of various sugars have been given as follows:—Lævulose, by Vosburg (*Analyst*, 1920, **45**, 383); invert sugar, by Zerban (*Analyst*, 1925, **50**, 294); inverted sucrose, by Monier-Williams (*S.P.A.*, 1928, **53**, 573); and

lactose, by Bacharach (*S.P.A.*, 1923, **48**, 521). For dextrin, see McLachlan (*S.P.A.*, 1928, **53**, 585).

INVERSION OF SUCROSE. Take a flask marked at 50 ml. and 55 ml. at 20° C. and put in 50 ml. of the sugar solution, add 3 ml. of 10E.HCl, mix and put in a water bath at 68–70° C. for ten minutes. Cool to 20°, and if the solution is 10 w/v, or less, dilute to the upper mark, but if 20 w/v dilute to 100 ml. Correct results were obtained with 2–20 w/v solutions of sucrose, and increasing the acid to 5 ml. and varying the temperature 63–76° C. made no difference, but the above conditions are recommended. Maltose and lactose showed no change in rotation when thus treated.

Boiling a 10 w/v solution of sucrose for ten minutes in N/500 HCl gave a solution with a rotation equivalent to 6.4 w/v. After allowing a 9.4 w/v solution of sucrose in N/2.HCl to stand cold all night, the rotation decreased, indicating 8.9 w/v.

The method advocated by the American Bureau of Standards differs from the above in that 5 ml. of 10E.HCl is added to the sugar dissolved in 75 ml. of water, and that it is always diluted to 100 ml. before polarising (cp. Monier-Williams, *loc. cit.*).

As the rotation of l  vulose is sensitive to temperature, becoming optically inactive at 87°, the temperature of polarisation must be carefully read.

REDUCTION, FEHLING SOLUTION. As there are several different strengths of this solution (Pegurier, *Analyst*, 1926, **51**, 91), care must be taken that the composition of the solution used in a particular process is that directed by the author. Lane and Eynon have pointed out the importance of using pure copper sulphate, and suggest that the solution should be standardised with invert sugar (*J.S.C.I.*, 1925, 150 T; *Analyst*, 1925, **50**, 244).

Elsdon has published detailed tables for use in the gravimetric determination of eight substances by Brown, Morris and Miller's method, which he gives (*S.P.A.*, 1923, **48**, 436, where there is a misprint in the strength of the copper sulphate solution, which should be 69.278 gm.). Bacharach (*S.P.A.*, 1923, **48**, 525) has investigated the gravimetric determination of lactose. It is advisable to do a blank on the Fehling solution itself and subtract any weight so found from the actual determination. The amount of sugar solution used should be sufficient to reduce most of the Fehling solution, or it may be difficult to wash all the unreduced copper sulphate from the filter paper.

For the volumetric determination Lane and Eynon have suggested methylene blue as an internal indicator (*J.S.C.I.*, 1923, 32 T; *Analyst*, 1923, **48**, 220; *S.P.A.*, 1924, **49**, 366). Ling and Carter praise the method and have suggested an improvement in it (*S.P.A.*, 1930, **55**, 730).

Lane and Eynon have found that the presence of calcium salts

gives low results in either the gravimetric or volumetric determination, and advise that they, together with any excess of lead used for clarifying, shall be precipitated with ammonium oxalate (*J.S.C.I.*, 1923, 143 T; *Analyst*, 1923, **48**, 277).

It should be remembered that oxygen interferes with the determination, and therefore boiled water only should be used for dilution, and that exposure to air during the determination should be as short as is possible.

In the following volumetric method the writer has avoided the use of any indicator by observing the colour of the filtered solution. If insufficient sugar solution has been added the filtrate will be blue; with a correct amount, colourless; and with excess, pale yellow.

Approximate determination. Heat 10 ml. Fehling solution with 40 ml. of water to boiling in a test tube (about 8 inch \times 1 $\frac{1}{4}$ inch) with pipe-clay. Add the neutral sugar solution, 1 ml. or less, at a time, till no apparent blue colour, then filter a little through 9 cm. Swedish filter. If the filtrate be blue, boiling with a little more sugar solution and again filtering will give an approximate figure. Dilute the sugar solution, if necessary, to between 0.5 and 1.0 w/v of reducing sugar.

Accurate determination. Put 10 ml. Fehling solution and 20 ml. of boiling water into a test tube as before. Measure the quantity of sugar solution, indicated by the approximate determination, into another test tube and add about 20 ml. boiling water. Put both test tubes in a water bath for five minutes, mix the contents, heat in water bath for five minutes, filter and observe the colour as before. Repeat, if necessary, with slightly different quantities of sugar solution. With weak sugar solutions the quantity of diluting water must be reduced so that the final volume is about 60 ml. With very dilute solutions less Fehling solution may be used. More accurate results may be obtained by putting 20 ml. or more of Fehling solution in a flask, but the amount of water must be proportionately increased.

Fehling solution. Make of B.P. strength; in making No. 2 solution it is a convenience to use a proportionate amount of 10E.NaOH, and it should be kept in a resistance glass bottle. It should be standardised. Weigh 4.75 gm. pure dry sucrose, dilute to 50 ml. invert as described above (p. 157), cool, neutralise and dilute to 100 ml. It will then be ten times the strength of Fehling solution.

Calculation.

If "a" gm. of sugar, or syrup, be diluted to "b" ml. (= 1)
and "c" ml. of (1) be diluted to "d" ml. by inversion (= 2)
and "e" ml. of (2) be diluted to "f" ml. (= 3)

and "g" ml. of (3) = 10 ml. Fehling solution = 0.05 gm. invert sugar.

and K = % (solids), or w/v (syrops), of reducing sugar expressed as invert sugar.

$$K = \frac{5 b d f}{a c e g}. \quad \text{If undiluted } K = 500/ag.$$

The equivalents of 10 ml. of Fehling solutions for other sugars are : sucrose (after inversion) 0.0475 ; maltose, 0.079 ; anhydrous lactose, 0.063 ; and the respective values of K are 105, 63, and 79, as compared with invert sugar, 100.

Effect of Sucrose. In the above conditions sucrose present in jam, treacle, etc., will have little or no effect on the determination of reducing sugar. When, however, 2 gm. of sucrose was present in the amount used in a determination, reduction was found to proceed at about the rate of 0.001 gm. in five minutes ; a pure sucrose would therefore show 0.05 % of invert sugar (cp. Beardsley and Bolton, *Q.J.P.*, 1929, 196).

Maltose. When the organic solids of a substance consist of maltose, with dextrose and lævulose only, the proportion of maltose may be calculated as follows :—

$$\text{Maltose, \%} = \frac{(\text{w/v of organic solids} - K) 100}{100 - 63}$$

FERMENTATION. Syrups and sugar foods usually contain large proportions of sucrose and invert sugar, and much smaller proportions of maltose, dextrose, dextrin, etc. It is obviously an advantage in analysis to concentrate the latter by removing as much as possible of the former by fermentation.

Weigh 20 gm. of the substance, add 220 ml. of water, if necessary filter out insoluble matter (pips, etc., in jam), add 1 gm. of yeast and sterilise by boiling a short time. After cooling add 3 gm. of yeast, plug with cotton wool and incubate at about 28° C. for five days, and a duplicate for seven days. Rotate flasks on third and fourth days. Decant 100 ml. of the nearly clear liquid, and measure the remainder (= "a" ml.). Evaporate the 100 ml. to less than

half to remove alcohol, add alumina cream, and dilute to $\frac{10,000}{100 + a}$ ml.,

so as to give a 20 w/v solution. After filtration, determine the reduction and rotation of the liquid. Dark-coloured solutions may require treatment with lead or chlorine, as mentioned above. A portion of the remainder from the fermentation is filtered and the organic solids determined by evaporating 10 ml. to dryness, drying the residue for six hours, igniting and subtracting ash.

The acidity of acid substances, as marmalade, should be reduced before fermentation. If the part fermented five days does not give

a satisfactorily low reduction ($K = 2$ or less, calculated on the substance), the seven days' fermentation should be proceeded with. If the rotation of the unfermented part is to the left, the K may be *assumed* to be invert sugar, if to the right dextrose; it has also been assumed that dextrin has no reducing power (cp. *S.P.A.*, 1928, **53**, 585). If the K is small, an incorrect assumption will have little influence on the final calculation. Examples of the calculations are given below under raspberry jam (p. 177). Matthews and Parker have given details of the fermentation of a sample of treacle (*S.P.A.*, 1900, **25**, 91). For differential fermentation by selected yeasts, see McLachlan (*S.P.A.*, 1928, **53**, 583).

CALCULATIONS AND CONSTANTS. There are ambiguities and discrepancies in published specific rotations; some authors do not state the temperature and concentration of the solutions used, whether it was neutral or acid, or whether the sugar was anhydrous or crystalline. With some difficulty the following values have been selected. Figures are given for the D sodium line ($\lambda = 5893$) and for the green mercury line ($\lambda = 5461$). The normal relation of the two values is, as 0.8492 to 1.0.

POLARISATION CONSTANTS OF SUGARS

Sugar.	Molecular Weight.	Sodium.		Mercury.	
		$10 \left[\alpha \right]_{D}^{20}$	A	$10 \left[\alpha \right]_{Hg}^{20}$	A
DEXTROSE, $C_6H_{12}O_6$. . .	180.09	52.7°	10.54°	62.1°	12.42°
LÆVULOSE, $C_6H_{12}O_6^*$. . .	180.09				
Solution neutral . . .		92.9°	18.58°	109.5°	21.88°
,, acid . . .		93.9°	18.78°	110.5°	22.1°
INVERT SUGAR, $2(C_6H_{12}O_6)^*$. . .	360.19				
Solution neutral . . .		20.1°	4.02°	23.7°	4.74°
,, acid . . .		20.6°	4.12°	24.2°	4.84°
SUCROSE (INVERTED), $C_{12}H_{22}O_{11}^*$. . .	342.17				
Solution neutral . . .		21.1°	4.22°	24.9°	4.98°
,, acid . . .		21.6°	4.32°	25.5°	5.10°
SUCROSE, $C_{12}H_{22}O_{11}$. . .	342.17	66.5°	13.3°	78.3°	15.66°
LACTOSE :					
Anhydrous, $C_{12}H_{22}O_{11}$. . .	342.17	55.2°	11.04°	65.1°	13.02°
Crystalline, $C_{12}H_{22}O_{11}$, H_2O . . .	360.19	54.2°	10.48°	61.9°	12.38°
MALTOSE :					
Anhydrous, $C_{12}H_{22}O_{11}$. . .	342.17	138.3°	27.66°	162.9°	32.58°
Crystalline, $C_{12}H_{22}O_{11}$, H_2O . . .	360.19	131.4°	26.28°	154.7°	30.94°
DEXTRIN, $n(C_6H_{10}O_5)$. . .	—	180°	36.0°	212°	42.4°

* = left-handed rotation.

A = rotation of 200 mm. 10 w/v solution at 20° C.

The above constants are for the standard temperature of 20° C., which should always be aimed at. Correction to it, for differences from it, may be made by the factors in the next table and the following equations.

Corrections, when the concentration differs from the standard 10 %, may be made similarly. Although the concentration is much

less under control than the temperature, corrections for the effect of concentration on the specific rotatory power are often trivial.

Apparently, the effect of acid on the temperature and concentration factors is inappreciable.

FACTORS FOR TEMPERATURE AND CONCENTRATION CORRECTIONS

Sugar.	B		b	C		Na and Hg.
	Na	Hg		Na.	Hg.	
Dextrose	0	0	—	·03	·04	·0005
Lævulose	·61	·72	·0065	·15	·18	·0008
Invert sugar	·31	·37	·015	·06	·07	·008
Sucrose (inverted).	·33	·39	·015	·06	·07	·008
Sucrose	·013	·015	·0002	·01	·01	—
Lactose, anhydrous	·077	·089	·0014	—	—	—
„ crystalline	·072	·085	·0014	—	—	—
Maltose, anhydrous	·095	·112	·0007	·02	·02	—
„ crystalline	·090	·106	·0007	·02	·02	—

B = decrease in numerical value of specific rotation for 1° C.

b = temperature coefficient.

C = increase in numerical value of specific rotation for increase of 1 w/v of sugar.

c = concentration coefficient.

The following related equations give the connection between specific rotation ($[\alpha]$), concentration (w/v) and angular degrees (α), when a 200 mm. tube is used. If the length be 100 mm., 100 should be substituted for 50 in the equations :—

$$(i.) [\alpha] = \frac{50\alpha}{w/v}. \quad (ii.) w/v = \frac{50 \alpha}{[\alpha]}. \quad (iii.) \alpha = \frac{w/v \times [\alpha]}{50}.$$

Temperature correction. This correction can never change the direction of rotation, and to avoid ambiguity, optical signs should be ignored. Equation (iv.), given below, is to be used when the specific rotation of a pure sugar is being determined ; equation (v.) when the actual angle of determination is being corrected for temperature, $[\alpha]$ may be substituted for α in this equation. The values of “ B ” and “ b ” are given in the previous table, and “ t ” represents ° C.

$$(iv.) [\alpha]^{20} = [\alpha]^t + B (t - 20). \quad (v.) \alpha^{20} = \frac{\alpha^t}{1.0 - b (20 - t)}.$$

The application of equation (v.) may be illustrated by the correction for temperature of a solution of invert sugar, which gave a rotation of -7.7° at 24.4° C.

$$\alpha^{20} = \frac{7.7}{1.0 + 0.015 (20 - 24.4)} = 8.24^\circ$$

Concentration correction. The standard concentration is 10 w/v. When a sugar solution is diluted, in addition to the primary reduction of the angle according to equation (iii.), there is, with some sugars, a

secondary reduction, owing to the fact that in a weaker solution the rotatory power *per unit of sugar* is less than in a stronger solution. the sign $^{10}[\alpha]$ means that the specific rotation has been corrected to 10 w/v for this secondary alteration. The corresponding sign for the angle actually observed in a 200 mm. tube is $^{10}\alpha$. Again, to avoid ambiguities, the optical sign of the sugar solution is ignored in making the correction.

Equation (vi.) is used for correcting the specific rotation to the standard 10 w/v, when the concentration is known. In many cases, however, the concentration of the solution has to be determined, and is unavailable. In such cases an approximation may be made by using the determined angle (which is related to w/v) instead of w/v, (vii.). The values for "C," "c," and "A" are taken from the tables :—

$$(vi.) \quad ^{10}[\alpha]^{20} = ^{\times}[\alpha]^{20} + C (10 - w/v).$$

$$(vii.) \quad ^{10}\alpha^{20} = ^{\times}\alpha^{20} + c (A - \alpha^{20})^{\times}\alpha^{20}.$$

The above-mentioned solution of invert sugar may be taken as an example of equation (vii.).

$$^{10}\alpha_D^{20} = 8.24 + 0.008 (4.12 - 8.24) 8.24 = 7.97^{\circ}.$$

Calculation of w/v when only one sugar is present. Either of the following equations may be used :—

$$(viii.) \quad \text{Sugar w/v in solution} = \frac{50 \times ^{10}\alpha^{20}}{^{10}[\alpha]^{20} \text{ of pure sugar}}.$$

$$(ix.) \quad \text{,, ,, ,,} = \frac{10 \times ^{10}\alpha^{20}}{A}.$$

With specific rotations the following equation may be used :—

$$(x.) \quad \text{Sugar (w/w) in the substance} = \frac{100 (^{10}[\alpha]^{20} \text{ of pure substance})}{^{10}[\alpha]^{20} \text{ of pure sugar}}.$$

Mixtures of sucrose and inverted sucrose. On inversion, 100 parts of sucrose yield 105.2 parts of invert sugar. "Inverted sucrose" is used to express invert sugar in terms of the sucrose, from which it is derived.

In recent years attention has been paid to the effect of acids and other electrolytes on the specific rotation of invert sugar (cp. *S.P.A.*, 1928, **53**, 573; 1930, **55**, 115). Probably owing to that cause, there is a considerable variation in the specific rotation attributed to that sugar, and consequently to those of inverted sucrose and lævulose, which are related to it. As inversion usually introduces acid, the "acid solution" constants of the previous table should be used. While it is believed that these constants are not far from the truth, it is advisable that each analyst should determine the change in the angle of a 10 w/v solution of pure sucrose *when using his usual method of inversion*. From this figure the other numerical values can be calculated. The angle after inversion must

be corrected for dilution, and for temperature if necessary ; the " change on inversion " is the sum of the two figures, each at 10 w/v and 20° C. Optical signs are ignored in the equations :—

$$(xi.) \quad ^{10}[\alpha]^{20} \text{ of invert sugar} = 5 \text{ (change on inversion)} - ^{10}[\alpha]^{20} \text{ of sucrose.}$$

$$(xii.) \quad ^{10}[\alpha]^{20} \text{ of invert sugar} = 0.95 \text{ } ^{10}[\alpha]^{20} \text{ of inverted sucrose.}$$

$$(xiii.) \quad ^{10}[\alpha]^{20} \text{ of lævulose} = ^{10}[\alpha]^{20} \text{ of dextrose} + 2 \text{ } ^{10}[\alpha]^{20} \text{ of invert sugar.}$$

The following equations refer to the calculation of sucrose :—

$$(xiv.) \quad \text{Angle due to sucrose} = 0.755 \text{ (change of angle of rotation).}$$

$$(xv. D) \quad \text{Sucrose, w/v in solution} = 1.135 \text{ (change of } ^{10}[\alpha]^{20}_D \text{).}$$

$$(xv. Hg) \quad \text{,, ,, ,,} = 0.934 \text{ (change of } ^{10}[\alpha]^{20}_{Hg} \text{).}$$

$$(xvi. D) \quad \text{,, ,, ,,} = 0.5675 \text{ (change of angle of rotation).}$$

$$(xvi. Hg) \quad \text{,, ,, ,,} = 0.482 \text{ (change of angle of rotation).}$$

In equations (xiv.) and (xv.) the change of rotation will be the sums of the angles if the sign of rotation is altered by inversion, and their differences if the sign is the same.

Equations (i.) to (xiv.) apply both to the sodium and mercury lines, but in most of the subsequent ones the factors differ, and the equations are given in two forms for the two lines.

As the addition of acid to inverted sucrose increases its specific rotation, the change on inversion may be somewhat too high, owing to this being added to the actual change due to the inversion of the sucrose. When the amount of inverted sucrose in the original solution is large, the calculated figure for sucrose may be somewhat too high. Neutralisation before polarisation does not prevent this small error, because ammonium chloride has a similar effect to hydrochloric acid (cp. Monier-Williams, *S.P.A.*, 1928, **53**, 573).

Comparison of the " angle due to sucrose " of previous equations with the angle before inversion will indicate if sucrose alone was present, or if there was also present a left- or right-handed polarising substance. The following two equations will only apply if it is known, or proved, that only sucrose and the products of its inversion are present.

The angle of the uninverted solution, corrected to 20° and 10 w/v if necessary, is indicated by " α ," and that of the solution after inversion corrected for the dilution of inversion, etc., by "I." The " — " sign is to be used if the optical rotation is changed by inversion. A negative result suggests dextrose.

$$(xvii.) \quad \text{Angle due to inverted sucrose originally present} = 0.245 \text{ } (3.07I \pm \alpha).$$

$$(xviii. D) \quad \text{Inverted sucrose, w/v, originally present} = 0.5675 \text{ } (3.07I \pm \alpha).$$

(xviii. Hg) Inverted sucrose, w/v, originally present = 0.482
(3.071 \pm α).

Sucrose and dextrose. The corresponding equations for mixtures of these sugars are given below; again “—” is to be used if inversion changes the optical sign:—

(xix.) Angle due to dextrose = 0.245 ($\alpha \pm 3.071$).

(xx. D) Dextrose, w/v, = 0.233 ($\alpha \pm 3.071$).

(xx. Hg) Dextrose, w/v, = 0.198 ($\alpha \pm 3.071$).

Lævulose and dextrose, invert sugar. Invert sugar is dextrose and lævulose present in equal quantities. If the quantities are dissimilar, a mixture of invert sugar with excess of either dextrose or lævulose results. A graphical method of calculation has been given previously (p. 108).

The following equations give the total amount of each (not the excess over invert sugar). The optical signs must be included as well as the algebraic ones:—

(xxi. D) Lævulose, % = 0.682 (52.7 — $^{10}[\alpha]_D^{20}$).

(xxi. Hg) „ % = 0.579 (62.1 — $^{10}[\alpha]_{Hg}^{20}$).

(xxii. D) Dextrose, % = 0.682 (93.9 + $^{10}[\alpha]_D^{20}$).

(xxii. Hg) „ % = 0.579 (110.5 + $^{10}[\alpha]_{Hg}^{20}$).

Correction for temperature and concentration of a mixture of two sugars. As sucrose and inverted sucrose polarise in opposite directions, the angle of a mixture of them is not a measure of the amount of inverted sucrose that is present. If it be advisable to make a correction, owing to the conditions differing widely from standard, an approximate calculation should first be made of the amount of inverted sucrose present in the solution before inversion, and the corrections calculated by using that approximate figure.

Mixtures of several sugars. When in addition to sucrose, more or less inverted, there is also glucose syrup present, it is necessary to supplement polarisation figures by determinations of reducing power and organic solids, before and after fermentation; or in some cases by making reasonable assumptions. An example will be found under jam (p. 178), and another of war-time damson jam has been given by the writer (*S.P.A.*, 1921, **46**, 449).

CHAPTER XIV

SUGAR, TREACLE, CONFECTIONERY, JAM, JELLY

White sugar, Demerara sugar. Treacle, golden syrup. Confectionery, chewing gum, stick liquorice. Jam. Marmalade. Jelly.

SUGAR

WHITE sugar is one of the purest articles in commerce ; crystal sugar rarely exceeds 0.02 % of moisture and 0.02 % of ash, and caster sugar is even purer. Only one of the 264 Birmingham samples of granulated sugar examined 1894–1930 was adulterated, and only one of the 182 samples of caster sugar examined 1911–30. In the latter case, two-thirds of the sample was ground rice, probably from an accident.

Demerara sugar, according to a memorandum of the West India Committee, is a sugar made chiefly in Demerara, Trinidad, St. Lucia, and British Guiana by a special process. No dye is used, the chloride of tin and phosphoric acid being used to fix the natural colour (*B.F.J.*, 1900, 175). There have been numerous prosecutions, with varying decisions, for the sale of cheaper “yellow crystals” as Demerara sugar. As “yellow crystals” is a distinct article of commerce, it would appear to be reasonable to expect that it should be, as in some cases it is, sold under its right name and not as “Demerara sugar.” The last decision I know of, in 1914, allowed considerable latitude in the interpretation of the word “Demerara sugar.” Apparently dyed beet sugar, if it can be detected, should not be sold as “Demerara.”

Of 109 Birmingham samples of Demerara sugar bought 1895–1900 only four were dyed. The usual composition varied from 0.2–0.5 % of ash, 0.3–0.8 % moisture, and 95–98 % of sucrose ; traces of tin were usually present, and fragments of sugar cane were often detected. Yellow crystals had a similar composition, but gave a bright red colour with strong hydrochloric acid. In a few cases sugar mites were found, which appeared as tiny floating yellow balls after the addition of water to the sugar.

The examination of fourteen samples of *sugar pieces* showed the usual composition to be 85–93 % of sucrose, with 0.3–8 % of invert sugar, 1.8–5.0 % of moisture, 0.7–1.1 % of ash.

Cassal gave a paper on “Dyed sugar” which was followed by a discussion. The colouring matter used for yellow crystals was stated not to exceed 1 in 50,000 (*S.P.A.*, 1890, 15, 141). Scard, in a letter

on the manufacture of Demerara sugar, stated that the amount of tin present was 0.25–0.33 grain per lb. (*Analyst*, 1890, **15**, 199).

During 1877–1889, there was practically no adulteration of “sugar” in England and Wales. Three later years, 1890, 1901 and 1913, had high figures, 13.8 %, 13.4 % and 10.8 %, respectively. The other years in the period varied from 1.3–9.2 % of adulteration. The large range may be due to varying proportions of white sugar and Demerara sugar being bought. During 1919–30, the average proportion had fallen to 0.6 %.

Ling has pointed out that raw cane products contain a smaller proportion of albuminoids (nitrogen \times 6.25) than raw beet products. Demerara sugar had 0.47 % against 0.89–1.35 % in similar beet sugar (*Jour. Inst. Brewing*, 1914, March).

PROSECUTIONS FOR SUGAR. *Birmingham.* Calcium carbonate in crystals 25 %. The purchaser was a lodging-house keeper. The Iceland spar was indistinguishable by appearance from the sugar with which it was mixed. Fine £5 (*F. & S.*, 1897, Jan. 30).

Edinburgh. Mineral matter 2.24 %, water 3.75 %, when it should have contained 99.5 % of sucrose. It was a soft yellow sugar. Evidence was given that at least four kinds of “sugar” were known to the sugar trade. Dismissed (*C. & D.*, 1901, June 15; *B.F.J.*, 1901, 243).

Northampton. Wheat semolina and possibly a little rice 13 %. Fine £5 (*Grocer*, 1918, March 23; *B.F.J.*, 1918, 56).

PROSECUTION FOR CASTER SUGAR. *Hayward's Heath.* Starch 11 %. It was said that ground rice had been accidentally added. Fine £2 (*Grocer*, 1912, March 2; *B.F.J.*, 1912, 80).

PROSECUTIONS FOR DEMERARA SUGAR. *Chippenham.* Dyed sugar 100 %, being “Yellow Crystals” and not Demerara sugar. Two vendors were each fined £1 (*F. & S.*, 1894, May 19).

Birmingham. Dyed sugar crystals 100 %. Fine £1 (*B.F.J.*, 1899, 217).

Birmingham. Dyed sugar crystals 100 %. Evidence was given by representatives of large firms of sugar brokers and manufacturers that in the wholesale trade “Demerara sugar” meant only sugar made in Demerara and free from foreign dye, and that Demerara sugar had a higher market value than raw sugar from any other source. The defence did not dispute these statements, but brought witnesses to prove that the custom of the *retail* trade was to sell any raw West India sugar, dyed or undyed, as “Demerara.” Case dismissed (*B.F.J.*, 1900, 143).

Tredegar. Crystals artificially coloured to resemble genuine Demerara sugar. Evidence was given by a Demerara Government analyst that the sample contained an artificial coal-tar dye and that dye was never used in Demerara. Fine £10 (*B.F.J.*, 1901, 66).

Clerkenwell. Cane sugar crystals coloured with an organic dye

foreign to genuine Demerara sugar. For the defence it was pleaded that "Demerara sugar" had become a generic term referring to a process of manufacture, and not to a place of production, and that the place of origin was immaterial. Evidence was given that it had been grown in Mauritius, and was equal to the best grade of West India sugar. Dismissed. On appeal, *Anderson v. Britcher*, the Court held that the magistrate had so stated the case that argument was hopeless, and in that particular case had properly found that the particular sugar was "Demerara." It was not to be understood, however, that any cane sugar coloured yellow might be called "Demerara." Appeal dismissed (*Analyst*, 1913, **38**, 595; *B.F.J.*, 1913, 154, 210; *Grocer*, 1913, July 26).

Lambeth. Dyed sugar other than Demerara. Evidence was given that the sugar had been imported from Demerara, and that at Greenock it had been washed, and then molasses and colouring matter added. After several days' hearing the magistrate came to the conclusion the summons must fail. There might have been a time when Demerara sugar was in fact sugar from Demerara, but that day had passed, and now "Demerara sugar" meant sugar of a certain quality and description. He considered the purchaser got as good an article as the article which was said by the Demerara people to be true Demerara sugar. He allowed the defendant 25 guineas costs (*Grocer*, 1913, Nov. 22, Dec. 13; 1914, Jan. 17, 31; *B.F.J.*, 1913, 227; 1914, 8, 32).

PROSECUTION FOR MOIST SUGAR. *London, Old Street*. Sea-sand $3\frac{1}{2}$ %. Fine £4 (*Grocer*, 1921, Sept. 3; *B.F.J.*, 1921, 100).

PROSECUTION FOR SUGARINE. *Stratford*. Milk sugar with 1.3 % of saccharin, and about seven times sweeter than loaf sugar. The wrapper stated it was twelve to fourteen times sweeter. Fine £1 (*Grocer*, 1918, Jan. 19).

TREACLE, GOLDEN SYRUP

Treacle is a dark-coloured syrup obtained during the refining of sugar, and there appears to have been no serious contention that it is anything but a sugar product. Some treacle is obtained by partial inversion of imported molasses.

"Golden syrup" is a term invented about 1840, and is applied to a syrup made from raw cane or beet sugar. After a single crop of sugar crystals has been obtained, the syrup is partly inverted by $\frac{1}{2}$ to 2 % sulphuric acid, the acid neutralised by chalk; the syrup is then filtered through animal charcoal and concentrated in vacuum pans (Arsenic Commission Report, II., 238).

About 1885 a practice of mixing glucose syrup with sugar syrup was introduced from America, and it has frequently been stated that the addition is necessary to prevent the sugar syrup from partly

crystallising. Lyle (*B.F.J.*, 1900, 38, from *Grocer*) contradicts this, and attributes the "graining" to lack of care in refining. With excess of lime salts, a dimness is produced in the syrup and the sugar may crystallise round a lime nucleus. Mixing dark treacle and colourless glucose syrup was an easy way of making a liquid resembling genuine golden syrup. Such mixtures may be legitimately sold as "amber syrup," "table syrup," "crystal syrup," "pale syrup," or "syrup." Unfortunately, the public does not recognise the fact that, as there is no standard for such articles, very inferior products may be sold under these names, even glucose syrup itself, the sweetening power of which is only about one-third of that of a sugar syrup. Some retailers, also, have failed to realise that articles invoiced to them under these names must not be sold as golden syrup.

In each of the two periods, 1900-13, 1920-30, 5.6 % of the samples of "treacle and syrup" examined in England and Wales were adulterated. Of the twenty-eight samples of golden syrup examined in Birmingham 1923-9, five were adulterated, and four of the thirty-two samples of treacle examined 1922-30. In 1923, owing to the purchase of adulterated samples, the Birmingham Medical Officer of Health sent a letter to the local Grocers' Association pointing out that the term "golden syrup" should be exclusively used for a cane sugar syrup, and that mixtures should be labelled as such. The Association endorsed this view (*Grocer*, 1923, May 5). Some labels used were not satisfactory. "Partly from sugar and partly from maize" was applied to an article of which only one-third was from sugar. The predominating constituent should have been named first. "A small proportion of glucose has been added" was not properly used when 40 % was there. Some of the worst samples, containing 90 %, or more, had been sold as "golden syrup" or "treacle"!

The following table is based on the analyses of about 100 Birmingham samples :—

ANALYSES OF TREACLE AND SYRUPS

	Golden Syrup.	Mixed Syrups.	Treacle.	Adulterated Treacle.	Glucose Syrup.	Glucose Chips.
Moisture	16-20	17-21	18-23	18-20	12-18	10-16
Ash	0.2-1.2	0.5-1.5	1-4	0.5-4	0.2-0.5	0.5-1.0
$[\alpha]_D^{20}$	13-16	35-100	12-19	35-110	112-119	44-48
Sucrose	31-36	3-25	28-36	3-30	0	0
Invert sugar	31-36	?	16-28	?	0	0
Reducing sugar (K)	38-45	32-48	33-45	34-43	31-43	73-84
UNFERMENTED RESIDUE						
$[\alpha]_D^{20}$	0 \pm 1	12-67	0 \pm 0.5	3-67	65-80	10-18
Organic solids—K	4-7	10-35	5-8	8-33	30-37	?

The term "sucrose" is preferred to "cane sugar," as it may have been derived from beetroot. Its proportion and that of invert sugar have been calculated from the polarisation figures before and after

inversion, on the assumption that no other optically active substance is present.

The writer has used the following formula for calculating the approximate amount of adulteration. As it is based on the assumption that sucrose and invert sugar are present in equal proportions, the amount will probably be underestimated :—

$$\text{Glucose syrup, \%} = \frac{[\alpha]_D^{20} - (0.7 \times [\alpha]_{20} \text{ due to sucrose})}{1.15}.$$

In doubtful cases it is desirable to proceed to fermentation, and calculate the composition, as subsequently indicated under jam (p. 177). As is shown in the table, glucose syrup is much higher in rotation and lower in reduction than glucose chips; its unfermented residue is also much higher.

A number of samples were tested for arsenic, but the amount found did not exceed 1 per million, except one sample of glucose chips obtained from a suspected source during the arsenic scare in 1900, when the amount found was over 500 per million.

Bodmer, Leonard and Smith have given analyses of a number of samples of golden syrup, and their methods of analysis (*S.P.A.*, 1899, **24**, 253). The *Analyst* for April, 1900, (pp. 85–98), has papers on the subject by Leonard, Jones, and Matthews and Parker, and the report of a discussion, which followed.

PROSECUTIONS FOR TREACLE. *Newton Abbot.* Glucose syrup 20 %. Fine 10s. (*B.F.J.*, 1902, 67).

Cromer. Water 30 %, being 12½ % in excess. Fine 1s. (*Grocer*, 1918, Sept. 14; *B.F.J.*, 1918, 106).

Nottingham. Glucose 80 %. Evidence was given that the article was sold as “table syrup,” and was labelled as such. Paid costs (*Grocer*, 1920, Jan. 24).

Nottingham. Starch syrup 80 %. Fine £5 (*Grocer*, 1920, May 8; *B.F.J.*, 1920, 50).

Halifax. Glucose syrup 60 %. Defendant said he had overlooked that the article was invoiced to him as “syrup.” Fine £2 (*Grocer*, 1930, Dec. 13; *B.F.J.*, 1931, 10).

PROSECUTIONS FOR SYRUP TREACLE. *Salford.* Corn Syrup 65 %. The label bore the words “Cane sugar with a percentage of corn syrup to prevent granulation.” Evidence was given that the term “Syrup treacle” was unknown in the trade. The stipendiary dismissed the case, as he considered the label was just sufficient, though it was deceptive, suggesting about 5 % of corn syrup. The manufacturers undertook to alter the label. In the second case a similar article was marked “Crystal syrup.” This case was also dismissed, the stipendiary remarking that if people want pure syrup, they must ask for “Golden syrup” (*Grocer*, 1923, Oct. 6; *Analyst*, 1923, **48**, 599, 600; *B.F.J.*, 1923, 98).

PROSECUTION FOR SYRUP. *Aberdeen.* Starch glucose syrup 90 %. Evidence was given that there were a great variety of syrups and the sheriff dismissed the case (*B.F.J.*, 1899, 281).

PROSECUTIONS FOR GOLDEN SYRUP. *Glasgow.* Starch glucose 20 %, or thereby, which is extraneous to golden syrup. It contained 27.6 % of cane sugar, 44 % of dextrose, maltose and fruit sugar, 11.8 % of dextrin and other organic matter, 0.7 % of mineral matter, and 15.9 % of water. The sheriff said it was clear that golden syrup was either a refiner's syrup or treacle which was obtained as a by-product in the refining of raw sugar, or a syrup prepared by the partial conversion (inversion) of either beet or cane sugar. Fine £1 (*B.F.J.*, 1899, 218).

Bristol. Glucose syrup 100 %. Fine £2 (*B.F.J.*, 1900, 27).

Liverpool. Glucose or starch sugar 70 %. Fine £5. On appeal to Sessions, the Recorder confirmed the conviction, considering that "Golden syrup" was a well-known name given to a certain article (*B.F.J.*, 1900, 102).

Worthing. Starch glucose 34 %. The Government analysts found 40 %. Fine £3 (*B.F.J.*, 1901, 172).

Southwark. Starch glucose 85 %. A manufacturer said that the golden syrup made by his firm was half invert and half sucrose sugar, and that starch glucose should not be present. Fine 5s. (*B.F.J.*, 1902, 18).

North London. Starch glucose syrup 68 %. It was stated that it was a common practice to sell an amber-coloured mixture of dark treacle and colourless glucose as golden syrup. Fine 10s. (*Grocer*, 1908, July 4; *B.F.J.*, 1908, 119).

West London. Glucose syrup 37 %. After several days' hearing the magistrate said he could not help thinking that golden syrup was a definite article and understood by the public to be a refined treacle. Treacle was understood to be the drainings from crystallised sugar in the course of its manufacture. Glucose, on the other hand, was not made from sugar. Fine £1 and 10 guineas costs (*Grocer*, 1910, July 16, 30; *B.F.J.*, 1910, 135, 151).

Wigan. Black treacle, containing 0.4 % of sand and dirt. The tin was labelled "Finest golden syrup, guaranteed pure." Fine 10s. (*Grocer*, 1912, Sept. 28; *B.F.J.*, 1912, 174).

Gateshead. Glucose not less than 70 %. It was said to be "crystal syrup." Fine £10 (*Grocer*, 1923, March 21).

London, Old Street. Glucose syrup 90 %. It was admitted that "amber syrup" was supplied. Fine £5 (*Grocer*, 1926, Jan. 15).

Stroud. The Inspector asked for a tin of "golden syrup," and after he had declared himself, the assistant said he had made a mistake and supplied "table syrup," which words appeared plainly on the tin. The magistrates dismissed the case, considering that before the sale was completed, the Inspector knew he was not buying

golden syrup. On appeal, *Miles v. Melias, Ltd.* (1930), the case was sent back to the lower court with instructions to convict. It was held that a cash transaction was contemplated, that when the goods were supplied the seller expected to be paid, and that there had been a sale. It mattered not that the money was not paid at the same second of time as the Inspector handled the syrup. On rehearing the vendors were fined £5 and 11 guineas costs (*Grocer*, 1930, March 29, May 10; *Analyst*, 1930, **55**, 326; *B.F.J.*, 1929, 106; 1930, 34, 56).

Hanley. "Crystal syrup" supplied instead of golden syrup. The amount of sulphur dioxide was also in excess, being 175 parts per million. Fine 2 guineas (*Grocer*, 1930, June 14; *Analyst*, 1930, **55**, 506).

CONFECTIONERY

About 1878 there were a number of prosecutions for sweets coloured yellow with the poisonous lead chromate. From about 1887 to 1897 paraffin wax appears to have been the popular adulterant. The Preservative Regulations of 1925 allow 70 parts per million of sulphur dioxide to be present in sugar and solid glucose, and 450 parts in liquid glucose. The amount of sulphur dioxide present in confectionery should not exceed the proportionate amount allowed for its constituents. Morgan has investigated the effect of temperature on the sulphur dioxide content in mixtures of sugars (*S.P.A.*, 1930, **55**, 488; 1931, **56**, 638).

In 1930 much illness, but fortunately no death, was caused by sweets containing 77–150 grains of arsenic per lb. Arsenic, left in premises which had been used for an enamel works, had been used for dusting sweets in the belief that it was French chalk! (*B.F.J.*, 1930, 103; *P.J.*, 1930, Sept. 27).

A curious sample of sweets was examined in Birmingham in 1931. It was in tablet form; the kernel was a chocolate mixture, next was a layer containing mineral matter, and outside was a coating of coloured sugar. It contained about 7 % of chalk and 3 % of talc. The manufacturer was cautioned and undertook to alter the composition of it.

Chocolate is considered later, under "Cocoa."

Of 265 samples of confectionery examined in Birmingham during 1900–27 only one sample was condemned, the adulteration being 0.6% of French chalk. Four of the eighty-five samples examined 1928–30 contained an excess of sulphur dioxide. Of those examined in England and Wales, 1927–30, 3.7 % were reported adulterated.

PROSECUTIONS. *Birmingham*. "Birds' eggs." In 100 there was present 3.7 grains of chromate of lead. Fine 5s. in each of three cases (1874 Report).

South Shields. Chromate of lead 0.68 % in the yellow portions. The vendor said that a man that he had employed instead of using vegetable colours, had used for the sweets yellow paint intended for painting a cart. Fine £5 (*Analyst*, 1879, 4, 216).

Newport, Isle of Wight. "Musk dessert." Brown sugar cubes contained $\frac{1}{7}$ grain of arsenic per lb. The arsenic was due to oxide of iron which had been added to make them look like chocolate. The magistrates dismissed the case on the defendant undertaking to inform the manufacturer of the objectionable nature of the coating (*B.F.J.*, 1903, 205).

Birmingham. "Woodbine cigarettes." Paraffin wax $5\frac{1}{2}$ %. Fine £2 (1896 Report).

Petersfield. "Jelly figs." Lead 15 parts per million. Fine £1 (*B.F.J.*, 1917, 108).

Wimbledon. "Sea beach shingle." Mineral matter, chiefly magnesium and aluminium silicate, 2.4 %. Talc had been used to dust the pans to prevent the sweets sticking. Paid 5 guineas costs (*B.F.J.*, 1921, 54).

Stockton-on-Tees. "Black currant and aniseed sweets." No trace of black currant. Another analyst found a small percentage. The Government analysts found no evidence of the presence of black currant tissue, or of the odour, taste or colour of black currant juice. According to the defendant, the process of manufacture would destroy all black currant tissue. The Bench dismissed the case, holding that there was no evidence to prove the absence of black currant juice (*Grocer*, 1925, March 14).

Bishop Auckland. "Rum and butter toffee." Butter fat 7 %, the remainder of the fat being coconut fat. The defence was that if butter only were used the toffee would become sticky and difficult to handle. Fine 50s. On appeal, *Riley Bros. v. Hallimond* (1927), the majority of the Court upheld the conviction, holding that toffee could be made from various fats, and that the word "butter" implied that no other fat was present (*Grocer*, 1927, May 21, Dec. 17; *Analyst*, 1928, 53, 93; *B.F.J.*, 1928, 4).

Stoke-on-Trent. A sugar boiler was fined £25 for powdering sweets with arsenic, and the manufacturer £15 for a similar offence (*P.J.*, 1930, Dec. 6).

Leicester. "Mint sweets." Sulphur dioxide in excess. The sulphur dioxide was stated to be used to make the sweets colourless and transparent. A full medicinal dose, for an adult, of sulphur dioxide was present in 2 oz. of the sweets. Three defendants were each fined £3 (*Grocer*, March 15, 1930; *B.F.J.*, 1930, 36).

Athenry. A small percentage of a silica drug. The justice said he had no evidence that the addition was harmful, and ordered the defendant to pay costs (*Grocer*, 1931, Sept. 26).

North London. "Black Currant Pastilles." Traces of artificial

colouring matter and synthetic flavouring, but no genuine black currant. The magistrate ruled that a purchaser would expect to get an article which contained some identifiable ingredient originating from the black currant. Fine 10s. (*Grocer*, 1932, Jan. 30; *B.F.J.*, 1932, 14).

CHEWING GUM

There have been cases in which large proportions of paraffin wax have been used in "chewing gum" and "chewing wax," and serious consequences have ensued to children who have eaten them, as paraffin wax is insoluble in the body. As the result of prosecutions makers marked the article "This is not to be eaten" or "You must not eat this," as if children, buying a cheap, attractive sweet, would attend to such directions, particularly when they were not very legible. Unfortunately, this labelling was held to put the article outside the Act, as neither a food nor a drug. In Birmingham, by the direction of the Public Health Committee, a circular was sent to the head teachers of schools asking them to warn their scholars against the sweets.

PROSECUTIONS. *Birmingham*. Paraffin wax 77 %. Fine 10s. (1888 Report).

Chesterfield. Paraffin wax 35 %. Case dismissed, as the label, "Not to be eaten," showed it was not sold as food. On appeal, *Shortt v. Smith* (1895), the High Court was unable to say that the magistrates were wrong (*F. & S.*, 1894, Dec. 22; 1895, April 6). *Bennett v. Tyler* (1900) was a similar appeal case.

Bradford. Pepsin chewing gum devoid of pepsin. Evidence given showed it was a mere matter of chance whether any particular packet contained any pepsin. As it had a label saying it was good for indigestion, the magistrate held that it was sold as a drug and fined the vendor £5 (*P.J.*, 1909, July 31; Aug. 7).

STICK LIQUORICE

Good Italian stick liquorice (Solazzi, etc.), which is sold for putting in linseed tea, under the name "Spanish juice," usually contains 20–30 % of matter insoluble in water and about 4–8 % of ash. It is a hard extract, while the "extract of liquorice" of the B.P. is a soft one. Detailed analyses have been given by Noble (*P.J.*, 1906, April 28).

In addition to the above, used as a drug, a mixture of liquorice with starch, etc., is sold as a sweetmeat, some of which has been branded "70" according to French law, which indicates 70 % of adulteration.

Bennett (*S.P.A.*, 1922, 47, 511) has called attention to the

substitution of an extract from a dangerous root, Masticogni, for liquorice (see also *P.J.*, 1923, Oct. 5).

PROSECUTIONS. *Southwark.* "Liquorice pipe." Starch and other foreign matter 50 %. The magistrate held that a pure extract, such as Solazzi, should be supplied when "liquorice" is asked for. The wholesale confectioner who sold it was fined £1 (*C. & D.*, 1892, Oct. 29).

London, North. "Liquorice." Added foreign starch, at least 25 %. The article was bought from a confectioner, and the stipendiary said the evidence showed that the liquorice of commerce and that used as a drug were two separate and distinct things; at the price at which it was sold he considered there had been no offence, and dismissed the case, allowing the defendant 2 guineas costs (*C. & D.*, 1893, April 1).

JAM

The position in relation to the composition of jam was altered considerably in 1930 by the publication of a memorandum by the Food Manufacturers' Federation of unofficial standards which had been arrived at in conjunction with the Society of Public Analysts (*Analyst*, 1930, **55**, 694; *B.F.J.*, 1931, 42). The standards were accepted by a large number of jam manufacturers, who also undertook to use the suggested labels and not to make any jam below the standards.

The "Full fruit standard" for single-fruit jams requires a minimum of 30 lb. (black currant) to 45 lb. (cherry or rhubarb) in 100 lb. of finished jam, and at least 40 lb. of fruit in mixed jams, the first-named fruit on the label being at least 20 lb. per 100 lb. of the finished jam. If there is too much fruit in a jam, it will be hard and will not spread. Jams of "Lower fruit standard" must have at least 20 % of fruit, and there must be at least 10 % of the fruit first named on the label. They must also be labelled "with other fruit juice," unless a mixed jam contains no added fruit juice or pectin.

To prevent mould, and to ensure that 1 lb. of jam shall go into a standard 1 lb. jar, the soluble solids of all jams shall be at least 68½ %. The addition of citric, tartaric and malic acid is permissible without declaration. The addition of such an acid is considered necessary when the acid in a fruit (*e.g.*, strawberry) is not enough to produce sufficient invert sugar, from the sucrose, to prevent granulation. The addition of colouring matter is also permitted, as some good quality jams may look unsatisfactory on keeping. No declaration of the presence of fruit juice is required for "Full fruit standard" jams. Some jams, as strawberry, cannot at times be satisfactorily made without it. The Jam (Sales) Order

of 1920 (revoked) permitted the addition of 10 % of added fruit juice.

These standards have been criticised, particularly in relation to the lack of declaration of additions (*B.F.J.*, 1931, 21, 31, 39, 51, 54). Whatever may be the defects in the scheme which time will show, an honest attempt has been made to obtain uniformity and to prevent the manufacture of grossly adulterated jam. The Council of the Society of Public Analysts has recommended that any jam containing less fruit than the full fruit standard sold without a suitable declaration shall be deemed to be adulterated (*S.P.A.*, 1931, 56, 427). Colonial standards have been given in the *British Food Journal* (1931, 41, 52, 61).

Samples of jam bought in Birmingham before the issue of the unofficial standards have shown considerable variation in composition. In various jams the water content has been usually 27–35 %, but a cherry jam was as low as 21.9 %, and a gooseberry jam as high as 38.6 %. The amount of sucrose was usually 21–33 %, but a sample of black currant had only 7.2 %, and one of gooseberry only 0.4 %, the sucrose having been nearly all inverted.

ADULTERATION. Of the samples of “jam” examined in England and Wales 1877–85, 6.3 % were adulterated, and 3.4 % of the samples of “jam and marmalade” examined 1927–30.

In spite of the fact that old-fashioned jam contained no glucose syrup, an appeal case, *Smith v. Wisden* (1901, p. 183) decided that 13 % might be used in marmalade, and after a long hearing in Glasgow, in 1905, 21.2 % was ruled to be not inadmissible in black currant jam. Glucose syrup was said to make jam set better and prevent crystallisation. In war-time the L.G.B. advised its use for jam-making, 1½ or 1¼ lb. being used instead of 1 lb. of sugar.

At one time apple pulp was a common adulterant, and the apple cells indicated its presence. Filtration was then resorted to for the removal of the tell-tale cells, not always with complete success, and there were convictions for the presence of apple matter (Marylebone, 1913, p. 181); see also a false warranty case at Bow Street (p. 181). In recent years pectin has been used to stiffen jam instead of apple or other juice. Food Investigation Report No. 33 of the Department of Scientific and Industrial Research (Review, *Analyst*, 1929, 54, 594) deals with the preparation and use of pectin. See also a paper by Back (*P.J.*, 1931, July 18). Agar-agar has also been used; for its properties and reactions, see Cooper and Nuttall (*P.J.*, 1908, May 23).

Some labels used for jam have been very deceptive. “Made from refined sugar” when glucose syrup was present. “Made from whole fruit” when no whole fruit could be detected. Some manufacturers attempted to protect themselves by labelling mixtures as “Improved (or, perfected) by the addition of fruit juice.” It

is notable that "improvement" was always with a *cheaper* fruit; never "Apple jam improved with raspberry," which would be a reasonable label. Further, some of the declarations of admixture were in such small type as almost to require a magnifying glass to read them. Other incorrect labels have stated the presence of a superior fruit which analysis has failed to detect.

In prosecutions for salicylic and benzoic acids in jam it has been stated that these preservatives enable less sugar to be used, and the boiling to be a shorter time; the effect of which has been that the jam contains an excess of water. There have also been prosecutions for excess of sulphur dioxide in jam; the Preservative Regulations limit the amount to 40 parts per million of jam. This limit has been found to be too stringent for black currant jam (*Analyst*, 1931, 56, 223).

ANALYSIS. Important papers have been communicated by Macara, who is the Director of Research of the British Association of Research for the Jam, etc., Trades (*Analyst*, 1931, 56, 35, 391, 701). They give the methods of analysis, and also analytical figures obtained with a large number of samples of different fruits. He gives a warning as to the necessity of samples of jam being thoroughly mixed before division for analysis.

In some prosecutions for raspberry jam, 3.0 % of insoluble matter has been taken as a standard for the calculation of the amount of raspberry present, and also the number of pips. Some Birmingham figures may illustrate the care necessary in using such figures. Five samples of "raspberry jam" contained sixty-three to eighty-one pips in 5 gm., while six samples in which the presence of fruit juice was declared had almost the same number—sixty-three to eighty-six. Six samples of raspberry and apple jam contained only six to thirteen pips in 5 gm. The average weight of a raspberry pip varied in five samples 1.2–1.7 mg. A sample of "bramble and apple" jam had on the average 1.4 pips per gm., but the top layer had nearly ten times the average, 10.1 pips per gm. The pips were so thick at the top that the purchaser brought it for analysis with a complaint. Three samples of black currant jam contained ten to eighteen currants in 26 gm., with 190 to 380 pips. Samples with fruit juice declared on the label had twelve to twenty-three currants, and one had 665 pips per gm. The practical difficulty of the uniform division of the insoluble matter makes it undesirable to form an opinion upon the analysis of a single pot of jam.

For the detection of glucose syrup, Judd Lewis has pointed out that the specific rotation of the saccharine solids of fruits is practically the same as that of invert sugar, and that polarisation after inversion of nine jams gave on that basis -20.5 to -22.9 , while with the addition of 10 % of glucose the figure was $+1.2$ (*S.P.A.*, 1930, 55, 384).

I am indebted to T. Macara for particulars of his method of calculating the proportion of glucose syrup in jam, which is shortly to be published in the *Analyst*. It is based on the assumptions that in fruits the dextrose and lævulose are present in equal proportions ; that the specific rotation (D) of the solids of glucose syrup is 140, and that reducing sugar, expressed as invert sugar, is half of them.

$$\text{Glucose solids, \%} = \frac{4K}{30} + \frac{2(J - S)}{3},$$

where K is the reducing power of the uninverted jam expressed as invert sugar, J is the specific rotation of the jam at 20°, and S is the corresponding figure for the sucrose in it.

The application of the formula to raspberry jam (I.), given below, indicates that it is genuine (actually — 0.07 % of glucose solids), while jam (II.) gives 8.7 % of glucose solids. This indicates 10.2 % of glucose syrup, on the assumption that 15 % of water is present in the syrup. The figure given in the table is “about 10 % of glucose syrup.”

In the tables below are given analyses of Birmingham jam examined by a more elaborate method, including fermentation.

The proportion of sucrose and the angle due to it are calculated from the rotations, by equations (xv.) and (xiv.), these figures and also those of the unfermented matter are subtracted from the original figures for organic solids, K, and specific rotation. If the resulting differences in organic solids and K are identical (as in jam I.), maltose is absent. If there be a notable difference (as in jam II.), the proportion of maltose can be calculated by the equation on p. 159. Subtracting maltose and its equivalent values will leave a remainder consisting of lævulose and dextrose ; the specific rotation of the mixture is calculated, and comparison with 20.6° (the rotation of invert sugar) will show if lævulose or dextrose is in excess. The one having the smaller proportion is calculated by equation (xxi.) or (xxii.), and doubling the result will give the proportion of invert sugar. This proportion is then applied to the actual w/v, and the difference expressed as (additional) lævulose or dextrose. If the unfermented matter has a left-handed rotation, the K of it is assumed to be invert sugar, and dextrose if right-handed. After the corresponding values have been subtracted, any right-handed rotation is assumed to be due to dextrin, and the residue after the subtraction of these is described as inert carbohydrates.

The maltose, dextrose and dextrin are added together to give glucose solids, and division by 0.85 gives glucose syrup. Comparing the two samples below, the larger proportion of unfermented matter in the one containing glucose syrup and the difference in the rotation of it are noteworthy. Experimental errors in a complicated analysis like this make it advisable to avoid giving an exact statement of the

amount of adulteration. In jam II. it was calculated to be 11·5 %, but was stated as "about 10 %." That was the usual proportion in Birmingham samples adulterated with glucose syrup.

ANALYSIS OF RASPBERRY JAM (I.)

	Organic Solids.	Reducing Sugar (K).	$[\alpha]_D^{20}$	Percentage of Jam.
After inversion			— 14·1°	
Before inversion	70·0	34·0	+ 14·8°	
Subtract sucrose	32·8	—	+ 21·8°	32·8
Difference	37·2	34·0	— 7·0°	
Subtract unfermented matter .	4·7	1·5	— 0·1°	
FERMENTED SUGARS	32·5	32·5	— 6·9°	
Lævulose and dextrose				
Calculate to 100	100		— 21·2°	
Dextrose	49·5			
Invert sugar	99·0			32·2
Additional lævulose				0·3
UNFERMENTED MATTER	4·7	1·5	— 0·1°	
Invert sugar	1·5	1·5	— 0·3	1·5
Difference	3·2	0	+ 0·2°	
Dextrin	0·1		+ 0·2	0·1
Inert carbohydrates	3·1		0	3·1
TOTAL				<u>70·0</u>

ANALYSIS OF RASPBERRY JAM (II.)

After inversion			— 0·8°	
Before inversion	73·5	35·0	+ 26·6°	
Subtract sucrose	31·2	—	+ 20·6°	31·2
Difference	42·3	35·0	+ 6·0°	
Subtract unfermented matter .	8·5	2·5	+ 7·3°	
FERMENTED SUGARS	33·8	32·5	— 1·3°	
Maltose	3·5	2·2	+ 4·6°	3·5
Lævulose and dextrose	30·3	30·3	— 5·9°	
Calculate to 100	100		— 19·4°	
Lævulose	49·2			
Invert sugar	98·4			29·8
Additional dextrose				0·5
UNFERMENTED MATTER	8·5	2·5	+ 7·3°	
Dextrose	2·5	2·5	+ 1·3°	2·5
Difference	6·0	0	+ 6·0°	
Dextrin	3·3		+ 6·0	3·3
Inert carbohydrates	2·7		0	2·7
TOTAL				<u>73·5</u>

The complete analyses of these jams were as follows :—

ANALYSES OF RASPBERRY JAM

Description :—	I. Made from best White Sugar only.	II. Made from Refined Sugar.
Water	27.0	22.9
Insoluble solids :		
Pips	1.9	2.1
Skin, etc.	0.8	1.2
Ash	0.3	0.3
Sucrose	32.8	31.2
Invert sugar	33.7	29.8
Lævulose	0.3	—
Dextrose	—	3.0
Maltose	—	3.5
Dextrin	0.1	3.3
Inert carbohydrates	3.1	2.7
TOTAL	100.0	100.0
No. pips from 5 gm.	76	63
Average weight of pips	1.2 mg.	1.7 mg.
Report on jam	Genuine	About 10 % of glucose syrup

For the detection of apple juice in jam, the papers of Muttelet (*Analyst*, 1922, **47**, 398 ; 1923, **48**, 181 ; 1927, **52**, 160, 598 ; 1928, **53**, 101) should be consulted. Partridge (*S.P.A.*, 1926, **51**, 346) recommends the use of the centrifuge and microscope for the detection of apple cells.

Carré and Haynes suggested the determination of pectin as calcium pectinate (*Analyst*, 1922, **47**, 263 ; 1923, **48**, 34). King has studied in detail the analysis of jams for pectin, agar-agar and gelatin, and the microscopical examination of hairs, etc. He has given illustrations of diatoms found in agar-agar (*S.P.A.*, 1925, **50**, 371). Parkes had previously reported on the detection of agar-agar (*S.P.A.*, 1921, **46**, 239).

PROSECUTIONS FOR JAM. *Blackburn.* Salicylic acid 2.0 grains per lb. The defendant's analyst found 1.05, and the Government analysts 2.1 grains per lb. Fine 10s. (*Grocer*, 1909, Nov. 20 ; *B.F.J.*, 1909, 208, 227).

Salford. Practically no whole fruit ; made from fruit pulp preserved with sulphur dioxide, and coloured with an aniline dye. It was advertised as being made from "Freshly gathered whole fruit, ripe and perfect." Fine £20 (*B.F.J.*, 1927, 48).

PROSECUTIONS FOR BLACK CURRANT JAM. *Southwark.* Almost entirely apple jam, with a little black currant as flavouring. Fine £2 (*B.F.J.*, 1903, 68).

Glasgow. Starch glucose syrup 21.2 %. Evidence was given by manufacturers of jam that glucose had been extensively used in jam for twenty-five to thirty years, and that though some fruits did not

require it, others were more easily made with it, and glucose prevented the jam granulating so readily. The sheriff dismissed the case on these grounds and considered the amount present was not inadmissible (*Grocer*, 1905, Oct. 28 ; *B.F.J.*, 1905, 222, 247).

London, Marlborough Street. Apple pulp at least 10 %. It was labelled "Improved with fruit jelly." The Government analysts confirmed the analysis. As fruit *pulp* was present, the magistrate fined defendant £12, but thought that if fruit *jelly* had been present, the label would have been a good defence (*Grocer*, 1911, March 18, April 20 ; *B.F.J.*, 1911, 55, 95).

Newcastle-on-Tyne. "It is impossible to state definitely the percentage of apple pulp, but I am of opinion that it forms a considerable proportion of the whole." Case dismissed as the certificate did not comply with the requirements of the Act (*Grocer*, 1913, Oct. 4 ; *B.F.J.*, 1913, 190, 214).

Widnes. Benzoic acid 5 grains per lb. Fine 10s. (*B.F.J.*, 1914, 173).

Driffild. Rhubarb 20 %, apple pulp 10 %. Fine £20 (*Grocer*, 1920, Sept. 18 ; *B.F.J.*, 1920, 94).

PROSECUTION FOR BLACK CURRANT AND APPLE JAM.

Newcastle-on-Tyne. Apple 50 %, gooseberry about 45 %, and black currant 5 %. The manufacturer stated that he had used 10 % gooseberry and 10 % black currant. As the manufacturer had taken means to rectify his error, he was only fined 5s. (*Grocer*, 1904, May 28 ; *B.F.J.*, 1904, 164).

PROSECUTION FOR BRAMBLE JAM. *Durham.* Apple 25% ; a quantity which was confirmed by the Government analysts. The defendants only admitted the presence of 10 %. Fine £2 (*B.F.J.*, 1900, 166).

PROSECUTIONS FOR DAMSON JAM. *Warrington.* Salicylic acid 2.1 grains per lb. The use of the preservative enabled more water to be put in the jam—39.7 % was present. Case dismissed (*Grocer*, 1906, March 17 ; *B.F.J.*, 1906, 95).

Omskirk. Benzoic acid $3\frac{1}{2}$ grains per lb. It was stated that less sugar and less boiling was required if benzoic acid was used. Fine 10s. (*Grocer*, 1914, March 14 ; *B.F.J.*, 1914, 53).

Heywood. Apple pulp 20 %. Fine 10s. (*Grocer*, 1930, May 3).

PROSECUTIONS FOR PLUM JAM. *Bury.* Apple pulp 25 %. An error in labelling was said to have happened. Fine £2 (*B.F.J.*, 1914, 128).

Newport Pagnell. Salicylic acid over 50 parts per million, and also fruit other than plum. The retailer was fined 25s. and the wholesaler £10 (*Grocer*, 1928, May 5 ; *B.F.J.*, 1928, 55).

PROSECUTIONS FOR RASPBERRY JAM. *Chester.* Rhubarb 50 %. Ordered to pay costs, as there was no fraudulent intent (*Grocer*, 1906, Dec. 15).

Ryde. Gooseberry, apple, raspberry, and sesame seeds, coloured with an aniline dye. Fine £1 (*Grocer*, 1910, Dec. 10 ; *B.F.J.*, 1910, 237).

London, Marylebone. Apple pulp at least 10 %. It was labelled "Choicest fresh fruit and refined sugar, improved with fruit juice." During three days, much expert evidence was called. The Government analysts found 2 % of apple pulp. The magistrate said that the evidence showed that for thirty to forty years standard jam had been regarded as jam containing the best fruit with refined sugar only, and that fruit juice had always been regarded as an adulteration. He considered the label was not sufficient to protect the seller, and that it was an inaccurate and misleading description, and calculated to secure an unfair advantage over other manufacturers. Fine £2 (*Grocer*, 1913, Nov. 15, 29, Dec. 13 ; *B.F.J.*, 1913, 230).

Belfast. Fruit juice, other than raspberry, 80 %. It was labelled "Special raspberry jam," which the manufacturers explained meant a quality *lower* than first-class. Fine £5 (*Grocer*, 1926, Jan. 15).

Waterford. Raspberry fruit fibre 1.74 %, being deficient in raspberry fruit by at least 40 %. Case dismissed ; the analyst's certificate did not say there was adulteration (*Grocer*, 1927, Aug. 6 ; *B.F.J.*, 1927, 90).

London, Bow Street. False warranty for jam containing at least 10 % of apple matter. A Government analyst found 0.49 % of pectin and a few apple cells. He estimated that 100 lb. jam would contain—raspberry fruit 20 lb., and fruit juice or pectin equivalent to apples not less than 24 lb. The insoluble solids in fresh raspberries varied 6–9 % ; he had calculated on 8.5 %. If fresh fruit were used, pectin was unnecessary for making jam, but old pulped raspberries required it. Evidence was given that the jam contained 40 % of raspberry, and 10–12 % of pectin, part of which the firm made by expressing and filtering the juice of cooked apples. It was stated that the first pots of jam filled contained the majority of the seeds, and the last pots very few seeds. The magistrate said the evidence showed that while apple juice gave consistence, it also lessened the expense, and might alter the flavour. He considered a purchaser ought to be informed of its presence, as some manufacturers did. Others made no disclosure as the public did not like it. Fine £20. Notice was given of an appeal, but it was abandoned (*Grocer*, 1927, June 18, 25, July 23 ; *B.F.J.*, 1927, 74, 84 ; *Analyst*, 1927, 52, 533). (*Note.* For a similar sample bought at Loughgall, see *Analyst*, 1927, 52, 535.)

London, Old Street. Foreign fruit pulp of the nature of gooseberry. Fine £20 (*Grocer*, 1928, March 3).

Londonderry. Raspberry fruit fibre 0.7 %, instead of at least 3%. The magistrates considered the label "Raspberry jam with

other fruit juice " was to conceal the inferior quality of the jam, and fined the defendant £10 (*Grocer*, 1931, Jan. 31 ; *B.F.J.*, 1931, 28).

London, Tower Bridge. Raspberry fruit 22 %, apple juice or pulp 21 %, gingly seeds 1 % (equal to 175 seeds per oz.). It was labelled " Pure raspberry jam, with a small amount of added fruit juice." The case was dismissed, a warranty being proved. The manufacturer was subsequently prosecuted for giving a false warranty. It was pleaded that it was a second-class jam, and that the " pure " on the label meant not injurious to health. Fine £10 and 10 guineas costs (*Grocer*, 1931, Nov. 21, Dec. 5 ; *B.F.J.*, 1932, 7).

PROSECUTION FOR GOOSEBERRY AND RASPBERRY JAM.

Stockport. Salicylic acid 2.6 grains per lb. It contained 32.9 % of water, while a genuine sample only contained 23.3 %. Fine 5s., which was confirmed on appeal to Quarter Sessions (*B.F.J.*, 1903, 208, 245).

PROSECUTION FOR RASPBERRY, GOOSEBERRY AND RED CURRANT JAM. *Bournemouth.* Gooseberry 70 %, raspberry 15 %, apple 15 %, and no red currant. Fine £1 (*Grocer*, 1906, July 21 ; *B.F.J.*, 1906, 172).

PROSECUTIONS FOR STRAWBERRY JAM. *Sunderland.* No whole strawberry, and at least 75 % of apple jelly. Fine £20 (*B.F.J.*, 1924, 54).

Gloucester. Apple flesh about 3 % of the sample, or 9 % of the fruit present. An analyst for the defence suggested that the three or four filtrations of the apple juice had not been sufficient to remove all the apple pulp. Case dismissed and defendants allowed 50 guineas costs (*Grocer*, 1927, Jan. 29).

London, Old Street. Strawberries deficient 64 %. It had been compared with the Food Manufacturers' Federation standard of 42 % of strawberries. The Public Analyst was not prepared to say that any apple juice was present. Evidence was given by the manager of the factory where the jam was made that no other jam was boiled at the same time ; he was unable to explain the deficiency. Case dismissed (*Grocer*, 1931, March 14, April 4 ; *B.F.J.*, 1931, 35, 45).

North London. Sulphite preservative (sulphur dioxide) 80 parts, in excess of the 40 parts per million allowed by the Preservative Regulations. Ordered to pay 3 guineas costs (*Grocer*, 1931, April 4 ; *B.F.J.*, 1931, 45).

London, Old Street. Other fruit jam 75 %. It was said that, owing to a mistake, " mixed fruits " jam had been sold. Fine 18s. (*Grocer*, 1931, May 9).

Drogheda. Tissues of apple fruit extraneous to strawberry jam. They were attributed to insufficient straining of the added apple juice. Fine 1 guinea (*B.F.J.*, 1931, 106).

MARMALADE

Eight of the nine samples examined in Birmingham in 1900-1 contained glucose syrup. Their acidity, expressed as citric acid, was 0.37-0.61 %. The sucrose varied from 16 % to 34 %.

Boseley (*S.P.A.*, 1898, **23**, 123) has given analyses of fifteen different makes of marmalade, and the methods of analysis used. Hanak (*Analyst*, 1930, **55**, 582) has given a method for the detection of carrots in marmalade.

PROSECUTIONS. *Worthing.* Starch glucose 13%. Fine £1, which was confirmed on appeal to Quarter Sessions. Evidence was given that for fifteen years glucose had been used in the manufacture of marmalade by a large number of, but not by all, manufacturers. Also, that its use prevented crystallising, and hindered mildewing and fermenting. On appeal to the King's Bench Division, *Smith v. Wisden* (1901), the conviction was quashed. There was no legal standard; a frequent, but not exclusive, use of glucose, and a variation of recipes. The article given to the purchaser, if different, was rather better. There was no evidence of inferior quality or of adulteration in the ordinary sense of the word (*B.F.J.*, 1901, 132, 160, 382; *Analyst*, 1901, **26**, 329).

Edinburgh Justiciary Appeal Court. *Wilson v. McCutcheon.* Starch glucose 14 %. The case had been dismissed by the sheriff-substitute, and the Appeal Court agreed with the decision as the complaint was lacking in definite specification; the article was a compound one for which no general standard existed. No opinion was given on the merits of the case (*B.F.J.*, 1902, 246).

Liverpool. Salicylic acid 5.5 grains per lb. Fine £5 (*B.F.J.*, 1904, 46).

JELLY

In 1930, seventeen samples of jelly tablets (raspberry and orange) were analysed in Birmingham. They showed a large variation in composition: water 1.3-33.3 %, gelatine (nitrogen \times 5.55) 5.8-14.5 %, sucrose 21.2-80.3 %, invert sugar 0.8-26.9 %. Five samples had no glucose syrup, the others had 5-30 %. Seven samples contained no sulphur dioxide, eight had 12-77, one had 140, and one 210, parts per million. All were below the amounts permitted by the Preservative Regulations in the constituents.

Of the samples of jellies examined in England and Wales in 1906-13, 5.2 % were adulterated, and 0.9 % of those during 1920-30.

PROSECUTIONS FOR BLACKBERRY JELLY. *Newcastle-on-Tyne.* Apple pulp at least 20 % of the fruit in the jelly, or 10 % of the whole sample. The case was dismissed as the jar was labelled "improved with rich fruit juice" (*Grocer*, 1915, March 13).

Newcastle-on-Tyne. Apple pulp 2 %. The magistrates held that

the label "Blackberry jelly and fruit juice" did not cover the addition of apple *pulp*. Fine £1 (*B.F.J.*, 1916, 300).

PROSECUTION FOR BRAMBLE JELLY. *Whitley Bay*. Apple jelly 90 %. Fine £10 (*Grocer*, 1924, Sept. 13 ; *B.F.J.*, 1924, 98).

PROSECUTIONS FOR BLACK CURRANT JELLY. *Greenwich*. Salicylic acid 5·6 grains per lb. Fine £1 (*Grocer*, 1912, March 9 ; *B.F.J.*, 1912, 46).

Bellingham. Apple pulp at least 3 %. Fine 5s. (*Grocer*, 1916, Feb. 19 ; *B.F.J.*, 1916, 279).

PROSECUTION FOR INVALID JELLY. *Southport*. Water 92·45 %, gelatine 5·06 %, mineral matter 0·44 %. The price of a pot containing 10 oz. was 1s. 3d. (*Grocer*, 1918, Dec. 7 ; *B.F.J.*, 1919, 8).

RED CURRANT JELLY. *Alnwick*. Largely, if not chiefly, gooseberry jelly. The jar had a label "Guaranteed pure red currant jelly." By microscopical examination, taste, and smell, the Public Analyst estimated 30–60 % of gooseberry was present. By the same tests the Government analysts reported that a large part of the sample was gooseberry jelly, but they were unable to define the proportion. Fine £3 (*Grocer*, 1913, May 3, 24 ; *B.F.J.*, 1913, 73, 100).

PROSECUTION FOR TABLE JELLY. *Blaina*. Salicylic acid 9·8 grains per lb. Case dismissed as vendor proved a warranty (*B.F.J.*, 1913, 139).

PROSECUTION FOR REAL FRUIT CRYSTALS. *Sunderland*. No trace of fruit juice, and coloured with a coal tar dye. The sugar present was entirely cane sugar. Evidence was given by the manufacturer that 10 oz. of fruit juice, and 4 oz. of real fruit essence, were used to each 40 lb. of the crystals. Case dismissed (*Grocer*, 1920, Jan. 31 ; *B.F.J.*, 1920, 16).

CHAPTER XV

MEDICINAL SYRUPS. HONEY AND PREPARATIONS

Easton's syrup. Syrup of phosphate of iron, chemical food. Syrup of rhubarb. Syrup of violets. Syrup of figs. Vinegar, syrup and oxymel of squill. Honey. Borax honey. Glycerin, honey and lemon juice.

MEDICINAL SYRUPS

THE amount of invert sugar in B.P. (1914) syrup, and the alteration on keeping, have been studied by Beardsley and Bolton (*Q.J.P.*, 1929, 196 ; *P.J.*, 1929, June 29 ; *C. & D.*, 1929, July 6).

EASTON'S SYRUP

Easton's syrup is a synonym for syrup of ferrous phosphate with quinine and strychnine. It is liable to oxidation, and the sp. gr. rises owing to the inversion of the sugar. Sp. gr. of the 1914 preparation about 1.27 (Evers and Caines, *Y.B.P.*, 1925, 406 ; see also, Timmis and Evers, *B.P. Conf.*, 1926, 427). It is suggested that in the 1932 B.P. the proportion of strychnine shall be halved.

Methods for the analysis of it have been given by Evers (*S.P.A.*, 1913, **38**, 447 ; *B.P. Conf.*, 1922, 409), Simmonds (*S.P.A.*, 1914, **39**, 81), Harvey and Back (*S.P.A.*, 1921, **46**, 188), and Haddock and Evers (*Q.J.P.*, 1931, 314 ; *P.J.*, 1931, July 25).

PROSECUTIONS. *Brighton.* Wilfully giving a false label "Easton's syrup. Poison," to an article devoid of strychnine. Fine £1 (*P.J.*, 1908, April 4 ; *B.F.J.*, 1908, 66).

Woolwich. Iron deficient 97.4 %. The wholesalers admitted sending to the retailer an article which was not B.P. syrup. Costs 2 guineas (*P.J.*, 1914, June 13 ; *B.F.J.*, 1914, 127).

Westminster. Applying a false trade description to an article containing no strychnine. The vendor was not a qualified chemist. The magistrates considered that the Food and Drugs Act would have been a more suitable one. Fine £2 and 8 guineas costs (*P.J.*, 1922, Jan. 7).

PROSECUTION FOR SYRUP OF IRON WITH QUININE AND STRYCHNINE. *Long Melford.* Quinine sulphate deficient 37 %. The wholesale dealers admitted sending to the retailer a different preparation. Paid costs, 11s. (*P.J.*, 1914, March 28).

SYRUP OF PHOSPHATE OF IRON

This article is required by the 1914 B.P. to contain 1 grain of anhydrous ferrous phosphate in one fluid drachm.

PROSECUTION. *Leighton Buzzard.* Deficient of 95 % of its proper strength. In answer to a question by the vendor, the Inspector said he would have the "white" kind. Fine 5s. and 22s. costs (*C. & D.*, 1898, Feb. 12).

COMPOUND SYRUP OF PHOSPHATES. CHEMICAL FOOD

A paper on the analysis and composition of trade samples has been given by Boa (*P.J.*, 1922, Jan. 28).

PROSECUTION. *Glasgow.* About $\frac{1}{3}$ grain phosphate of lime and about $\frac{1}{4}$ grain iron, per teaspoonful, while it was represented to contain $2\frac{1}{2}$ grains phosphate of lime and 1 grain of iron. Fine £3 (*Analyst*, 1881, 6, 52).

SYRUP OF RHUBARB

According to the 1914 B.P., this syrup should contain 84 w/v of sucrose, and as the rhubarb will probably yield about 3 w/v of soluble matter, the total solids should be about 87 w/v. No less than five of the twelve Birmingham samples examined 1928-9 were below this figure :—

SOLIDS IN SYRUP OF RHUBARB

Total Solids, w/v .	76.3-77.9	80.6-82.2	87.5-89.0	89.9-91.3	Total
No. of Samples .	2	3	4	3	12

The sugars were determined by polarisation ; in nine samples dextrose was present (0.8-3.7 w/v), in two invert sugar (1.3, 1.9 w/v), and one sample contained neither.

SUGARS IN SYRUP OF RHUBARB

AMOUNT, w/v .	70.8	72-	74-	76-	78-	80-	82	Total
NUMBER OF SAMPLES :								
Sucrose .	1	3	2	-	6	-	-	12
Total sugars .	-	2	2	2	1	3	2	12

It is evident that most of the sugar was in the form of unchanged sucrose.

SP. GR. OF SYRUP OF RHUBARB

Sp. gr. .	1.28-	1.30-	1.32-	1.33, 1.34	Total
No. of samples .	3	2	5	2	12

PROSECUTION. *Birmingham.* Water 20 %. Fine £3 (1894 Report).

Bingley. Excess water 23 %. Dismissed on warranty being proved (*F. & S.*, 1897, May 15).

SYRUP OF VIOLETS

The London Pharmacopœia directed that it should be made from fresh violet petals, sugar water and spirit, but none of the twelve samples examined by Kendall were genuine (*P.J.*, xiii., 19).

PROSECUTION. Syrup coloured by violet aniline dye. The maker admitted that it was syrup flavoured with essence of violets and coloured with aniline dye. Case dismissed (*C. & D.*, 1902, July 19, 26 ; *B.F.J.*, 1902, 182).

SYRUP OF FIGS

PROSECUTIONS. *Dromara*. Salicylic acid 0.04 %. Fine and costs 16s. (*B.F.J.*, 1928, 120).

Toomebridge. Salicylic acid 0.194 %. It was argued that as "compound syrup of figs" was not a standardised preparation, no offence had been committed. Summons dismissed (*P.J.*, 1929, Feb. 28).

Eglington. Salicylic acid 0.01 %. The summons was dismissed as the article was described as a food and not as a drug (*Grocer*, 1931, Jan. 10 ; *Analyst*, 1931, 56, 106).

VINEGAR OF SQUILL

In 1902 considerable interest was aroused by the prosecution in London of three vendors for selling vinegar of squill deficient in acetic acid (*P.J.*, 1902, May 3, 10 ; *B.F.J.*, 1902, 163). The prosecutions were noteworthy as being based on a deficiency in strength of the solvent, without any suggestion that the articles were of inferior medicinal value.

According to the 1898 B.P. the soluble matter of 12.5 gm. of squill was to be contained in 100 ml., 4.27 % acetic acid being used as the solvent. The Public Analyst allowed 0.07 % for loss, etc., and used as his standard 4.2 % of free and combined acetic acid. Evidence was given for the defence that the properly prepared article might contain from 3.3–3.9 % of acetic acid. A vendor whose sample contained 2.5 % was fined 10s. and costs, but another prosecution for 2.58 % was dismissed. The third sample was very muddy and contained fungoid growth, with only 1.56 % of acetic acid. The Public Analyst's certificate contained no remark about decomposition ; the explanation he gave was that any decomposition would not interfere with the analysis, and the magistrates considered this explanation as satisfactory, but dismissed the case, as the vendor only sold the article under the compulsion of the Inspector.

At the invitation of the editor of the *Chemist and Druggist*, a number of pharmacists gave their experience of the vinegar of squill (*C. & D.*, 1902, May 24). The figures given indicated that the usual

range of composition was : acetic acid 3.6–3.9 w/v, total solids 7–9 w/v, sp. gr. 1.033–1.043. Bell (*loc. cit.*) examined a sample of squill, and found 68.8 % of it to be soluble in dilute acetic acid, and 19.2 % to be insoluble, the remaining 12.0 % being moisture.

The lower acetic strength of the vinegar than of the acid used in making it has been attributed to formation of acetates from calcium salts or other chemical change, or to evaporation. The latter explanation is negatived by Chattaway (*S.P.A.*, 1903, **28**, 29), who found that vinegar of squill, like acetic acid, became *stronger* on evaporation.

Cripps (*P.J.*, 1907, 519) found that this preparation lost only 0.1 % of acetic acid in three years. A sample made by the writer had sp. gr. 1.039, total solids 8.8 w/v ash, 0.33 w/v, and 3.7 w/v of acetic acid. The latter after 2½ years had fallen to 3.5 w/v.

In the 1914 B.P. the proportion of squill was practically doubled, but in the 1932 B.P. it is to be reduced.

SYRUP OF SQUILL

This preparation, if made according to the 1914 B.P., should contain 65.0 % of sucrose and at least 1.13 % of acetic acid. If the vinegar of squill used in preparing it contain 18 w/v of total solids, the syrup should contain 3.2 % of squill solids. Seven Birmingham samples examined in 1929 were in reasonable agreement with the B.P. requirements, the range of constants being as follows : sp. gr. 1.33–1.34, acidity 1.06–1.16 % acetic acid, total solids 67.4–69.6, $[\alpha]_D^{20}$ before inversion 13.8 to 38.2, and after inversion – 14.5 to – 16.5. From the latter figures the sucrose varied 33.2–61.8 %, and the invert sugar 12.4–39.5 %. The latter figures, of course, include any optically active matter derived from the squill. In each of the samples more or less inversion of the sucrose had taken place. One sample was condemned ; it contained only 50.7 % of solids, and the other figures were proportionately low.

Clark has given (*Y.B.P.*, 1911, 300) particulars of the examination of four samples of the 1898 preparation, which was very similar to that of the 1914 B.P. An alteration is suggested for the 1932 B.P.

OXYMEL OF SQUILL

The B.P. of 1914 requires that its sp. gr. shall be 1.29, with acidity equivalent to at least 1.43 % of acetic acid. Ten Birmingham samples examined in 1928 were in reasonable agreement with these standards, having sp. gr. 1.28–1.34, and 1.2–1.7 % acetic acid. The total solids varied from 59.1 % to 62.5 %, and $[\alpha]_D^{20}$ from – 4.8 to – 10.5 ; four samples were below the B.P. limit of – 7.8 at 15.5° and six samples above it. These figures support Franklin's

statement that the B.P. limit is too low ; he suggested — 11.0. Little alteration in strength is suggested for the 1932 B.P.

One sample taken from a wholesale supply gave the following figures : sp. gr. 1.30, acetic acid 0.8 %, $[\alpha]_D^{20}$, before inversion + 58.5 and after inversion + 57.3. It had been prepared with glucose syrup instead of honey. It differed from most of the genuine samples in being clear instead of slightly turbid.

According to F. Browne (*P.J.*, 1928, May 12) the colour of the oxymel depends on the amount of heating used in purifying the honey. He quoted reports showing that the deposit is free from medicinal activity, and composed of fat, pollen grains, and mould fungus spores.

PROSECUTION. *London, Marlborough Street.* Prepared with sugar instead of clarified honey. Fine £2 and costs (*P.J.*, 1908, May 9 ; *B.F.J.*, 1908, 85).

HONEY

The following figures depend on the analyses of forty-eight samples bought in Birmingham, 1915-31 :—

WATER IN HONEY

Water, %	15-	16-	18-	20-	22-22.8	Total
Percentage of samples	17	33	29	13	8	100

SPECIFIC ROTATION OF HONEY

$[\alpha]_D^{20}$ (left-handed)	5.3-	8-	10-	12-	14 to 15.9	Total
Percentage of samples	8	15	40	29	8	100

SPECIFIC ROTATION OF HONEY AFTER SUBTRACTION OF ROTATION OF SUCROSE

$[\alpha]_D^{20}$ (left-handed)	8.5 to 9.3	10.2-	12-	14-	16 to 18.3	Total
Percentage of samples	4	36	33	23	4	100

SUCROSE IN HONEY (BY POLARISATION)

Sucrose, %	0-	1-	2-	3-	4-5.5	7-8.9	Total
Percentage of samples	19	31	21	15	10	4	100

The ash of forty-four of the forty-eight samples was under 0.3 %. In eighteen samples that had been successfully fermented, the reduction (K) of the residue being 1.2 % or less, the $[\alpha]_D^{20}$ varied from + 0.1 to + 2.1.

Detailed analyses of honey have been given by Browne and Young (*Analyst*, 1908, **33**, 358), Witte (*Analyst*, 1910, **35**, 61), Lendrich and Nottbohm (*Analyst*, 1912, **37**, 53), Fiehe and Stegmüller (*Analyst*, 1913, **38**, 265), and Caulkin (*P.J.*, 1927, May 14), who found that some presumably genuine West Indian honeys had $[\alpha]_D^{20}$ as high as — 24. For the range of composition of Canadian

honey, see Shutt (*Analyst*, 1932, **57**, 36). Frost has given an interesting account of bee-keeping and products (*P.J.*, 1902, Feb. 1, 8, Nov. 8, 29).

ADULTERATION AND ANALYSIS. Very few samples of honey have been reported adulterated in England and Wales; during 1905-13 the proportion was 0.4 %, and in 1920-30, 0.6 %. Only one of the forty-nine samples examined in Birmingham 1915-30 was adulterated.

Adulteration may be by excess of water, by addition of cane sugar (sucrose), glucose syrup, or invert sugar as artificial honey.

For water, Witte (*Analyst*, 1911, **36**, 276) suggests a limit of 20 %, while the limit of the United States Department of Agriculture is 25 % (*B.F.J.*, 1930, 73). The highest Birmingham sample, as is shown above, was 22.8 %. A usual commercial standard is that a gallon shall weigh at least 12 lb., but good samples may weigh 14 or 15 lb. (*Grocer*, 1920, March 6).

The amount of sucrose in normal honey is not large. In seventy-two samples of Russian honey, Sarin found from 0 to 5.5 % (*Analyst*, 1913, **38**, 149); Zoneff states that Bulgarian honey has usually less than 3 % (*Analyst*, 1927, **52**, 598), while sixty-three samples from various countries examined by Lendrich and Nottbohm (*Analyst*, 1912, **37**, 53) ranged 0.04-5.36 %. Witte (*opus cit.*) gives 6 % as a limit, and the U.S. Department of Agriculture (*opus cit.*) 8 %. The question is complicated by the possibility that, in a bad honey season, bees may visit sugar or molasses factories. Frost (*opus cit.*) traced the cause of some of his honey being pink or red to a two-way traffic of his bees between the hives and a toffee works. Sometimes bees are intentionally fed on sugar syrup, but honey so produced is flavourless and should not be considered to be normal honey (Witte, *opus cit.*). In 1929 the supplier of a Birmingham sample of "honey" attributed the 40 % of sucrose present to his bees! Published figures indicate that such an amount of sucrose has been added *outside* the hive, and not by bees. Sarin (*opus cit.*) was unable to distinguish five of his samples yielded by sugar-fed bees from the others. Samples of honey made by bees living near a sugar refinery had 12.6 % of sucrose (Bensemann, *Analyst*, 1888, **13**, 75) and 3.9-16.4 % (Von Lippman, *Analyst*, 1889, **14**, 20). There is the further question that the nectar of flowers is largely sucrose, which is inverted by a ferment in the body of the bees. In an experiment by Korndorfer (*P.J.*, 1911, Oct. 21), bees were fed on a 50 % solution of sucrose, and it was found that in the four minutes which elapsed between them filling and emptying their honey bags, four-fifths of the sucrose had been inverted, and in a few days only 1 % of sucrose remained. Achert (*Analyst*, 1912, **37**, 194) found that honey itself, if not heated above 55° C., inverted sucrose. In four months, the 22 % present in a mixture became

reduced to 2.2 %. For honey diastase, see Braunsdorf (*Analyst*, 1931, **56**, 539) and Fiehe (*Analyst*, 1931, **56**, 540). H. H. Bagnall was informed by bee-keeping authorities that honey from sugar-fed bees would not contain more than a small percentage of sucrose, and that, in any case, none should be present in honey sold to the public (Birmingham Analyst's Report, 1929). Bees do not contain formic acid (Merl, *Analyst*, 1922, **47**, 76).

For the determination of glucose syrup the writer has used the following formula :—

$$\text{Glucose syrup, \%} = \frac{([\alpha]_D^{20} \text{ of non-sucrose} + 12) 100}{115 - (-12)}$$

The following table gives figures obtained with samples which had been adulterated in the laboratory. It will be observed that only part of the sucrose added was detected ; this may be due to sucrose being inverted by the honey, as mentioned above :—

ANALYSES OF ADULTERATED SAMPLES OF HONEY

$[\alpha]_D^{20}$	Original Honey.	10 % Sucrose added.	10 % Glucose Syrup added.	10 % Sucrose and 10 % Glucose Syrup added.
Honey	-11.4	-5.9	+0.6	+6.7
After inversion	-12.0	-13.3	-0.7	-0.7
Due to sucrose	+0.5	+5.6	+1.0	+5.6
Due to non-sucrose	-11.9	-11.5	-0.4	+1.1
After fermentation	+1.1	+0.6	+3.2	+3.1
REDUCTION (K)				
Honey	75.1	67.0	71.5	63.1
After inversion	75.2	77.6	72.4	73.6
ORGANIC SOLIDS AFTER FERMENTATION, %				
SUCROSE, CALCULATED %				
By polarisation	0.7	8.4	1.5	8.4
By reduction (K)	0	10.1	0.9	10.0
GLUCOSE SYRUP, calculated by formula above, %				
	0.1	0.4	9.1	10.2

As honey is largely invert sugar, the addition of perfectly pure invert sugar could not be detected ; it is therefore necessary to test for some impurity in commercial invert sugar as an indication of adulteration. Analyses and standards for artificial honey have been given by Behre (*Analyst*, 1919, **44**, 237 ; 1921, **46**, 499) and Beythien (*Analyst*, 1921, **46**, 500). The tests of Ley (*Analyst*, 1907, **32**, 291) and Fiehe (*Analyst*, 1908, **33**, 397) have been frequently used (see *Analyst*, 1909, **34**, 399 ; 1910, **35**, 434 ; 1911, **36**, 451). Fiehe's test and others have been exhaustively examined by Lampitt, Hughes and Rooke, as well as the effect of heat, and the presence of diastase in honey (*S.P.A.*, 1929, **54**, 381 ; 1930, **55**, 666). Auerbach and Bodländer, by oxidation with iodine, have determined the ratio between fructose and glucose for the detection of artificial honey (*Analyst*, 1924, **49**, 389 ; see also 1925, **50**, 191). Atkins has determined lævulose in honey by oxidation with bromine (*S.P.A.*, 1917, **42**, 12).

General methods of analysis have been given by Fiehe and Stegmüller (*Analyst*, 1912, **37**, 449), and methods for the determination of dextrin by Lucius (*Analyst*, 1926, **51**, 581 ; 1927, **52**, 599), and Fiehe (*Analyst*, 1909, **34**, 440). Fabris has examined various methods for determining water in honey (*Analyst*, 1911, **36**, 586). Microscopical examination for pollen grains may be advisable. (For the determination of honey in honey cake, see Mees, *Analyst*, 1929, **54**, 108.)

Invert Sugar. The usual composition, as deduced from twelve Birmingham analyses, was : water 15–22 %, ash 0.2–2.0 %, sucrose 0–3 %, and the $[\alpha]_D^{20} = 4$ to -12 . Three samples after fermentation had 8–11 % of organic solids, of which 2–3 % was K ; and specific rotation -0.8 to -1.6 .

PROSECUTIONS. *Central Police Court.* Glucose 57 %. Fine £2 (*Analyst*, 1878, **2**, 166).

Pontypridd. Dextrose, or starch sugar, 50 %. Fine 71s. (*F. & S.*, 1896, Aug. 15).

Banff. Sugar 45.2 %, being in excess of 10 %—the maximum found in genuine honey. Case dismissed on warranty “Guaranteed pure Scotch clover honey.” The wholesaler was subsequently fined £5 for giving the false warranty (*Grocer*, 1913, Feb. 12, April 7 ; *B.F.J.*, 1913, 72).

Marlborough Street. Applying false trade description “Pure Cambridgeshire honey” to an article which microscopical examination showed was foreign honey. Fine £10 (*B.F.J.*, 1915, 99).

Aberdeen. Non-invert sugar 34.6 %, instead of a maximum of 8.6 %. The defendant’s bees were said to have been fed on sugar. Fine £5 (*B.F.J.*, 1917, 17).

Gateshead. Water 37 %, dextrin 14.65 %, while the water should not exceed 25 %, and there should be no dextrin. It was labelled “Delicious flavoured Table Honey.” Fined £10 (*B.F.J.*, 1919, 76).

East Dereham. Starch syrup and cane syrup 70 %, and sweetened with saccharin, not more than 30 % of genuine honey. Fine £1 (*Grocer*, 1920, Nov. 6).

West London. Applying false trade description, “Heather honey,” to an article which did not contain pollen grains derived from heather. Fine £20 (*Grocer*, 1927, March 19, April 9 ; *Analyst*, 1927, **52**, 340 ; *B.F.J.*, 1927, **36**, 48).

BORAX HONEY

According to the B.P., borax honey should contain 10 % of borax, the other ingredients being clarified honey and glycerin. The following figures relate to Birmingham samples examined in 1915 or 1928–31 :—

BORAX IN BORAX HONEY (Sixty Samples)

Borax, %	2.8-8.3	8.5-	9.5-	10.5-	11.5-14.0	Total
Percentage of samples . .	11	20	37	23	9	100

The fact that only 37 % of the samples were within 0.5 % of the theoretical figure cannot be considered satisfactory, and a number of vendors were cautioned. A vendor who sold two samples which contained no honey was prosecuted. The upper clear part of a sample that had deposited contained 11.5 % of borax and the lower turbid part 9.2 %.

SPECIFIC ROTATION (Fifty-five Samples)

$[\alpha]_D^{20}$	2.9-	6.0-	8.0-	10.0-12.2	Total
Percentage of samples . .	18	29	33	20	100

LOSS ON DRYING IN WATER OVEN (Forty Samples ;
0.5 gm. ten hours)

Loss on drying	20.7-	22-	24-	27-	30-	33-34.1	Total
Percentage of samples . .	10	32	25	10	15	8	100

In these conditions borax loses part of its water. The sp. gr. usually varied from 1.37 to 1.42. In seven samples the sucrose, determined by change of rotation on inversion, varied 0.7-1.7 %.

ANALYSIS. An approximate determination of the borax can be made by direct titration, but previous ignition is more satisfactory ; the addition of soda being unnecessary. Multiplication of the percentage of ash by 1.89 gave in most cases a good approximation to the amount of borax present.

Before polarisation the liquid must be made acid ; in one sample, the specific rotation without the addition of acid was + 0.9, but when acid to litmus - 0.5, and when acid to methyl orange - 5.6 (cp. Levy and Doisy, *Analyst*, 1930, **55**, 50).

PROSECUTION. *Birmingham.* Borax 32 %, glycerine 68 %. Evidence was given that the vendor asked the Inspector to call in an hour's time for the article. Fine 10s. (1930 Report).

GLYCERIN, HONEY AND LEMON JUICE

PROSECUTION. *Bradford.* " Glycerin trace, lemon juice trace. As this sample is relatively free from glycerin and lemon juice . . . I am of opinion that it is not of the substance, nature and quality demanded by the purchaser, and it is therefore adulterated." The Public Analyst, in evidence, admitted that there was no standard, and suggested that 30 % of glycerin was a reasonable one. For the defence it was claimed that the certificate disclosed no offence, as the Public Analyst had not put his standard on it. After an adjournment the case was dismissed for that reason (*Grocer*, 1930, April 12 ; *B.F.J.*, 1930, 47, 56).

CHAPTER XVI

MILK. CONDENSED MILK. DRIED MILK. BUTTERMILK

Average composition. Range of composition. Monthly variation. THE APPEAL TO THE COWS. Range of composition. Yield and composition. Relation of fat to solids-not-fat. Intervals between milking. Feeding. Variation in composition from day to day. Effects of cold weather. Excessive drinking by cows, rainfall. CONCLUSIONS AS TO LIMITS. Probability of mixed milks below limits being genuine. ADULTERATION. Toning. Rain. Rising of cream. Distributors. Statistics. Profits of adulteration. PROSECUTIONS. Added water. Deficiency of fat. Starch, salt, etc. ANALYSIS. Total solids. Fat. Sp. gr. Freezing-point, etc. Normal relation of constituents. Calculation of milk adulteration. FORE MILK. SEPARATED MILK, skimmed milk. SOUR MILK. PRESERVED MILK, formalin. COLOURED MILK. HEATED MILK, bottled milk, scald milk. DIRT IN MILK. CONDENSED MILK. DRIED MILK. BUTTERMILK.

OWING to the size of the subject, little more can be given than Birmingham experience. Besides special books on the subjects, reference may be made to the Departmental Report, and Minutes of Evidence, on Milk and Cream (1901) ; to the Report and Minutes of the Scotch Inter-departmental Committee of 1922 ; to Tocher's " Variations in the Composition of Milk " (1925) ; to the Ministry of Agriculture and Fisheries' " Variations in the Composition of Milk " (1929) (Miscellaneous Publications, No. 65) ; to a criticism of the latter by the Committee of Public Analysts (*Analyst*, 1929, **54**, 472) ; to numerous papers by Richmond (*S.P.A.*, 1892-1920) ; and to papers by Monier-Williams (*S.P.A.*, 1920, **45**, 203), and Tocher (*S.P.A.*, 1926, **51**, 606).

AVERAGE COMPOSITION OF BIRMINGHAM MILKS, 1874-1930

Period.	1874-	1879-88	1893-	1903-	1913-	1923-30
Fat %	—	—	3·63	3·55	3·60	3·61
Solids-not-fat %	—	—	8·61	8·70	8·72	8·77
Total Solids	11·0	11·9	12·24	12·25	12·32	12·38

As all samples are here included, adulterated as well as genuine, the results are lower than would be given by pure milk. Owing to alteration in methods of analysis, the total solids only can be given for the first two periods. These low figures are due to adulteration ; in 1881 the L.G.B. Report pointed out Birmingham as being conspicuous for milk adulteration. Subsequent to that period, except for a slight improvement in solids-not-fat, the figures were remarkably constant.

RANGE OF COMPOSITION. Averages are unsatisfactory for indicating the proportions of adulteration ; the following classifications give more information :—

RANGE OF COMPOSITION OF MILK, 1893-1930

PERIOD :	1893-	1903-	1913-	1923-30
FAT.				
Under 3 %	10	7	5	4
3.0-	29	37	31	31
3.5-	37	40	43	45
4.0-	15	13	17	16
4.5-	9	3	4	4
	<hr/>	<hr/>	<hr/>	<hr/>
	100	100	100	100
SOLIDS-NOT-FAT.				
Under 8.0 %	8	3	2	1
8.0-	11	4	4	2
8.3-	9	8	10	7
8.5-	59	66	66	64
9.0-	13	19	18	26
	<hr/>	<hr/>	<hr/>	<hr/>
	100	100	100	100
PERCENTAGE OF ADULTERATION	15.7	9.9	8.8	5.9
TOTAL SAMPLES	3,963	8,847	20,869	20,248

In the first period, 10 % of the samples were below the limit for fat, and 28 % below the limit for solids-not-fat. In the last period these figures had fallen to 4 % and 10 % respectively. The proportion of samples containing 9.0 % of solids-not-fat and over, had doubled, but there was no corresponding improvement in samples of milk containing 4.0 % of fat and over, probably owing to the mixing of milks during pasteurisation producing a more uniform richness. The proportion of adulteration fell from 15.7 % to 5.9 %.

Owing to the variation in the richness of milk, vendors have assumed that a good milk may be watered without fear of detection ; in some cases this is correct. One farmer when prosecuted, stated, " I know 2 gallons of water in 17 of milk will pass, for I have it on good authority." His trust in the " good authority " cost him £20 fine.

The method of determination of total solids has varied very little, and an interesting comparison of three periods is given on p. 196.

In the first period 12 % of the samples were badly watered, containing under 10 % of total solids, in the middle period 2 %, and in the last year less than 1 %. On the other hand, the good samples containing over 12 % of total solids increased from 33 % in the first period to 69 % in the second and 81 % in the last year. The improvement may be more strikingly shown by means of an arbitrary scale. If 1 black mark be given for every per cent. of samples between 11 % and 12 % of total solids and 2 black marks

THE APPEAL TO THE COWS

The decision in *Hunt v. Richardson* (see p. 40) that milk sold "as it came from the cow" is genuine, whatever may be its composition, has made this appeal very important. A farmer is now to be "tried by a jury of his own cows."

In Birmingham, as has been previously stated (p. 23), it has been the practice to invite farmers, whose milks have been found to be below the presumptive limits, to allow inspectors to visit the farms, see the cows milked, and take samples of the milk.

During twenty-one years, 1905 to 1925, 216 different farms, in eight counties, have been visited, and 434 samples taken. In each case the farm was visited because defective milk had been sent to Birmingham; the results, therefore, are not averages, but an indication of what cows *cannot* do in the quality of their milk. Poor milks have been obtained by selection and good milks only when the farmer has watered his milk. A large proportion of the cows were of the shorthorn breed. More than a quarter of the samples were taken in war-time, when the cows were badly fed.

It was the duty of the inspectors to ask that the cows should be milked in the usual way, and in some cases they noted that the strippings were unfairly divided between different churns. For this reason, where more than one churn of milk has been sampled at a farm, the average composition of the whole meal, paying attention to the quantities of the different fractions, has been used as the unit in the calculations given below. Cows, obviously, cannot be blamed for a farmer's carelessness in mixing strippings.

The number of cows at the farms varied from three to fifty. The recorded composition of milk from *single* cows shows large variation. Richmond ("Dairy Chemistry") found 4.9-10.6 % of solids-not-fat, and Tocher (*op. cit.*) 7.0-10.66 %, while for fat, the latter found 1.66-7.50 %, Richmond 1.04 %, and Hodgson 19.5 % (*S.P.A.*, 1923, 48, 443). For practical purposes, variations in the milk of individual cows is of little importance, as milk sent in by rail or road is not from one cow, but from a herd.

RANGE OF COMPOSITION OF FARM MILKS

PERCENTAGE OF SOLIDS-NOT-FAT

Under 8.3	8.3-8.4	8.5-8.9	9.0 and over
6 %	14 %	68 %	12 %

PERCENTAGE OF FAT

Under 3.0	3.0-3.4	3.5-3.9	4.0 and over
3 %	23 %	32 %	42 %

Although the presumptive limit for solids-not-fat is 8.5 % it has been my practice to pass samples of milk containing 8.3 % as

genuine if there is a fair amount of fat. The worst sample was one which contained 8.1 % of solids-not-fat and 2.9 % of fat. It was taken from the milk of three poorly-fed cows whose total yield at the milking was only 1 gallon and cannot be taken as normal milk. Only 5 % of the Birmingham supply farms numbered as few as three or four cows. The next worst samples came from a farm where the evening milking gave 8.2 % of solids-not-fat, and the morning 2.8 % of fat ; the other figures were not below the limits. The herd was in poor condition and contained some old cows ; the intervals of milking were thirteen and a half and ten and a half hours. These were the only two farms from which samples low both in solids-not-fat and fat were obtained.

Morning samples from six other farms were below the limit for fat. In each case the evening milk was of good quality and the mixture of the morning and evening milk was above the limit. The chief cause of the low fat was the long interval from the previous milking. In five of the farms from fourteen to fifteen and a half hours had elapsed since the evening milking. At one of the farms there were only three cows, and in two cases the cows were in poor condition.

YIELD AND COMPOSITION. The average yield from the farms for the two meals was 1.93 gallons per day. It is sometimes suggested that the milk from cows giving a good yield is poor. The following table shows that the quality of the milk is little affected by the amount yielded :—

YIELD AND COMPOSITION OF FARM MILKS

AVERAGE COMPOSITION	GALLONS PER DAY			
	Under 1.5	1.5-1.9	2.0-2.4	2.5 and over.
Solids-not-fat .	8.56 %	8.66 %	8.74 %	8.78 %
Fat . . .	3.88 %	3.69 %	3.64 %	3.64 %
Total Solids .	12.44 %	12.35 %	12.38 %	12.42 %

RELATION OF FAT TO SOLIDS-NOT-FAT. It might be expected that milks high in fat would be low in solids-not-fat, and *vice versa*, but, on the contrary, good milk tends to be high in both.

RELATIONS OF FAT AND SOLIDS-NOT-FAT IN FARM MILKS

Average percentage of solids-not-fat.	Less than 8.5	8.5-8.8	8.9 and over
Average percentage of fat . . .	3.51	3.67	3.97

INTERVALS BETWEEN MILKING. The following comparisons are from farms that were visited morning and evening and when the same number of cows were milked for each meal :—

YIELD. COMPOSITION. MORNING AND EVENING 199

AVERAGE COMPOSITION AND HOURS FROM PREVIOUS MILKING, FARM MILKS

Meal interval-- hours	Evening.			Morning.		
	9-	10-	11-12	12-	13-	14-15
Solids-not-fat .	8.62 %	8.59 %	8.69 %	8.74 %	8.73 %	8.74 %
Fat .	4.16 %	4.07 %	3.71 %	3.48 %	3.51 %	3.39 %
Total Solids .	12.78 %	12.66 %	12.40 %	12.22 %	12.24 %	12.13 %
Differences :—						
Solids-not-fat						
Morning–Evening	0.12 %	0.14 %	0.05 %	—	—	—
Fat						
Evening–Morning	—	—	—	0.23 %	0.56 %	0.77 %

Evening milk is richer in fat than morning milk, and the percentage of fat increases with a decreased interval. With similar intervals, eleven to twelve hours, the evening milk contained 0.23 % of fat more than the morning, but when the intervals were nine-, and fourteen-fifteen hours, the evening milk was 0.77 % higher in fat.

On the other hand, the differences between the amounts of solids-not-fat were trivial :—

SOLIDS-NOT-FAT IN MORNING AND EVENING FARM MILK

Averages.	Fat %.	Solids-not-fat %.	Total Solids %.
67 Morning meals .	3.46	8.74	12.20
67 Evening meals .	4.04	8.63	12.67
314 meals .	3.84	8.67	12.51

The above averages of the morning meals are 0.48 % of fat less, and 0.11 % of solids-not-fat more than those of the evening meals.

Stock (*S.P.A.*, 1930, 55, 541) has investigated the effect of alteration of milking hours on fat, and found that there was no definite mathematical relation between them.

It has been suggested that if cows are milked three times a day, a notable difference is made in the proportion of solids-not-fat. Experiments to test this were made by the courtesy of a local farmer who allowed his cows to be milked at irregular intervals under the observation of our inspectors.

Fifteen cows were milked at 4.50 p.m. on the first day, three times on the second day, at 7.10 a.m., 1 p.m., and 7.20 p.m., and at 6.50 a.m. on the third day. The solids-not-fat only varied by 0.3 %, but, as was expected, there was a considerable difference in the percentage of fat. The lowest figure, 3.4 %, was obtained at a morning milking after an interval of nearly twelve hours, and the highest fat, 5.6 %, at a mid-day milking, the interval being nearly six hours.

When the cows were fetched up for milking at the unusual time of 1 o'clock, they appeared uneasy, and instead of going straight to their respective stalls, had to be driven there. Although they

had been milked at 1 o'clock they came to the gate at their usual time, about 4.30, and waited there till they were milked at 7.20. In spite of the interference with the cows' regular habits the quality of the milk was hardly affected ; the following results were obtained :

EFFECT OF MILKING THREE TIMES A DAY

	Fat %.	Solids-not-fat %.	Gallons.
Averages : two meals	4.27	8.75	27
three meals	4.27	8.87	26
Range	3.4-5.6	8.7-8.9	

The small differences show that the extra labour required for milking three times a day, was practically wasted.

FEEDING. The position has been recently summarised ("Variations in the composition of Milk," p. 12), as follows : "It remains broadly true to say that where cows are suitably fed milk cannot be altered appreciably in respect of the percentage of butter fat by the foods, and that as regards the solids-not-fat the effects are still more difficult to trace." The 1922 Scotch Report (*op. cit.*) made a similar remark and continued : "Underfeeding, we are advised, unless carried to an extreme point, may result in a diminished quantity of milk, but not in an alteration of quality."*

When the Birmingham inspectors visited farms they classified the conditions of the cows as "poor," "fair," or "good." The following averages represent the mixed morning and evening milk from each farm :

CONDITION OF COWS IN RELATION TO QUALITY AND YIELD OF MILK

Condition of Cows.	Solids-not-fat.	Fat.	Total Solids.	Daily Yield.
Poor	8.52 %	3.47 %	11.99 %	1.77 gallons.
Fair	8.59 %	3.64 %	12.33 %	1.72 "
Good	8.80 %	3.77 %	12.57 %	2.13 "

It will be seen that the good cows yield on the average about 0.3 % of solids-not-fat and fat more than the poor cows, and about one-third of a gallon per day more than the other two classes. These results support the view that insufficient feeding of the cows will affect the quantity of the milk more than the quality and also that the condition of the cows will suffer before there is any serious drop in the quality of the milk.

Several farms sending milk containing less than 8.3 % of solids-not-fat were visited by the veterinary surgeon, who reported that the low proportion was due to the cows being insufficiently fed, or to the food having a low nitrogenous value. In a number of cases the cows were in poor condition owing to improper feeding during war-time.

* See Addenda, p. 577.

In a more recent case the evening milk from a farm contained 8.0 % of solids-not-fat, and 3.5 % of fat. A veterinary surgeon reported that the condition of the cows was very bad, and due to insufficient nourishment, and that if the conditions continued it would not only be a case of stopping the milk, but also of cruelty to animals by starving them.

The farmer was advised to give each cow at least 6 lb. of good cake per day, and three weeks later, the solids-not-fat had increased to 8.8 %. It is often alleged that the composition of milk containing a considerable excess of water is due to the starvation of the cows, but in this case, though the starvation appears to have been extreme, the milk was only 0.5 % below the presumptive limit of 8.5 %.

A farmer managed to persuade the Birmingham magistrates that his defective milk was due to the cows being moved from old turf to aftermath grass, which he stated produced an increased quantity of milk of poor quality. The accuracy of this novel defence was tested on six other cows. They had been fed on old turf for a month, and after milking were taken to a field upon which aftermath had been growing for about a fortnight. The following results were obtained :—

EFFECT OF FEEDING COWS ON AFTERMATH

Date.	Fat %.	Solids-not-fat %.	Gallons.
Sept. 2nd, old turf . .	4.8	8.6	about 6
„ 4th, aftermath . .	4.4	8.8	„ 6
„ 6th „ . .	4.2	8.6	„ 6
„ 9th „ . .	4.5	8.6	„ 5

It will be seen that neither was the quantity increased nor the milk made poor by the change ; the farmer's defence was probably more remarkable for its ingenuity than for its accuracy.

VARIATION IN COMPOSITION FROM DAY TO DAY. While the milk of a single cow may show considerable variations from day to day (cp. Richmond, *S.P.A.*, 1903, **28**, 290), the larger the number of cows in a herd, the more will this individual variation be minimised. If herds of cows frequently show large daily variations, the “ appeal to the cow ” is not only futile but misleading. The 1922 Scotch Report (*op. cit.*) stated : “ There is not much variation, from week to week, in the content of milk from a herd, and we consider the appeal to the milk of the herd should be regarded as evidence in any case of actual adulteration that may arise, if it is carried out within seven days after the official sample is taken.”

In Birmingham the practice has been to connect the offending sample with that taken at the farm by daily samples (p. 22). Evidence is thus obtained upon which an opinion can be formed as to whether any improvement in the milk is due to the cessation of

addition of water, or to variability in the cows. Milks from two farms give an illustration of this :—

DAILY MILK SENT FROM A FARM

Date of Sampling	Nov. 22	24	26	29	Dec. 6	7	8	9
Solids-not-fat %	8.05	8.3	8.45	8.15	7.95	7.95	8.7	8.85
Fat %	3.5	3.6	3.65	3.55	3.2	3.3	4.0	3.85
Gallons, about	28½	28	28	28	27	27	25½	24½

Informal samples were taken on the first four days, the farmer received an intimation by post on Dec. 7th, *after* his milk had been sent. The improvement in quality and the diminution in quantity was immediate; he attributed the adulteration to his cowman, and was fined £10.

The figures for the milk from the second farm are a contrast :—

DAILY MILK SENT FROM A FARM

Date of sampling	April 2	19	24	25	26	27
Solids-not-fat %	8.1	8.0	8.05	8.05	8.2	8.05
Fat %	3.75	3.2	3.1	3.15	3.1	3.4
Gallons	—	—	28	27	27	26½

The first two samples were informal; the farmer knew on the 25th that his milk was being sampled, but little alteration resulted. He was cautioned and instructed to feed his starved cows better.

Sometimes in prosecutions, a change in composition of the milk, which has been aptly described as “lightening,” has been alleged, perhaps as the result of an addition to a single meal of the cows. Remarkable intelligence appears to be attributed to some cows, who not only recognise the presence of an inspector, but also repent of their sins of omission, and produce immediately a milk of good quality!

Figures have been published (“Variations in the Composition of Milk”) for the milk of a dairy of twenty-four cows which showed considerable fluctuations in quality; the writer believes that such fluctuations are very exceptional, and it should be noted that, in spite of the fluctuations in composition, not one of the thirty-nine samples was below the presumptive limits for genuine milk. It by no means follows that similar variations above *and below* the limit for solids-not-fat occur.

On three occasions a practical test has been made of the actual day-to-day variations of the milk sent to Birmingham. The farms were in different parts of the country, and were selected for convenience of daily sampling. The milking was not supervised, and the farmers knew nothing of the experiment till it was completed. The first farm was sampled in January, 1926, for eleven days. The mean temperature varied from 27°–46° F., and snow fell on three days; in spite of the unstable temperature the solids-not-fat only

varied from 8·7–8·9 %. In the following August, another farm was sampled on sixteen days, when the solids-not-fat varied 8·6–9·3 %. The third experiment was made by my successor (H. H. Bagnall) in October, 1929, over sixteen days. He remarked that the farm was on a hill in a very dry place, and that the drought preceding the sampling had made conditions of feeding and watering very poor. Also: "The weather included every variety that the British climate at its best can provide, and the temperature varied from 37°–65°. The cows, therefore, had every opportunity of upsetting all calculations. As a matter of fact, however, the quality of the milk during the period was extraordinarily constant as regards the solids-not-fat, and even the notoriously variable fat percentage did not provide any great day to day variation." The solids-not-fat only varied from 8·5–8·9 %. After the experiment it was ascertained that the first herd consisted of twenty-two cows, the second of seventeen cows in the evening and sixteen in the morning, and the third of twenty-six cows in the morning and twenty-two in the evening. Fore milk was sometimes present in the first case, so that farm could not be used for fat comparisons. In the third farm six calves were put to their mothers.

The milk supplied by a retailer was also tested daily from January 4th to February 7th, and the solids-not-fat varied from 8·4–9·2 %. I have no information that the milk was obtained from the same cows each day.

For convenience of comparison the total results of the samples from the three farms have been tabulated and the differences calculated to percentages.

VARIATIONS IN DAILY SAMPLES OF MILK

Differences between meals.	FARMERS.			RETAILER.		
	One day.	Two days.	Three days.	One day.	Two days.	Three days.
SOLIDS-NOT-FAT.						
0, 0·1 % . .	76	70	64	57	35	39
0·2 % . .	15	20	25	20	24	36
0·3 % . .	6	8	9	7	28	18
0·4 % . .	3	1	2	13	10	0
0·5 % . .	0	1	0	3	3	7
	<hr/>	<hr/>	<hr/>	<hr/>	<hr/>	<hr/>
	100	100	100	100	100	100
FAT.						
0–0·2 % . .	65	53	49	63	51	57
0·3, 0·4 % . .	22	35	39	17	28	11
0·5, 0·6 % . .	9	6	9	17	14	29
Over 0·6 % . .	4	6	3	3	7	3
	<hr/>	<hr/>	<hr/>	<hr/>	<hr/>	<hr/>
	100	100	100	100	100	100
OBSERVATIONS .	76	70	64	30	29	28

Considering the great variation in conditions and temperature, it is remarkable that only about 10 % of the comparisons of farms show more than 0·2 % of difference in solids-not-fat, even when three

days have intervened. Also, that only about 13 % of the fat comparisons give more than 0.4 % difference.

The variations in fat in the retailer's samples are not large, which shows that milk can be kept mixed during retail sale ; part of this variation may be due to the milk on different days not being from the same cows.

In 1930, daily samples were taken at the Lancashire County Council's farm for periods of about two and four months, respectively. One herd was sampled from July to November, and G. D. Elsdon reported that "the solids-not-fat were practically constant throughout the whole of this time, whilst the variations of fat were unimportant." The other herd was similar (County Analyst's Report, 1930).

In forty-four instances Birmingham inspectors visited farms in the evening and also the next morning :—

SOLIDS-NOT-FAT IN EVENING AND MORNING FARM MILKS

Excess of solids-not-fat in morning milk over that of previous evening.										
%	0.5, 0.4	0.3	0.2	0.1, 0	-0.1, -0.2	Total.
Percentage of samples	9	11	16	55	9	100

It will, therefore, usually be to the advantage of a farmer, if the adulteration of a morning sample be calculated on the amount of solids-not-fat found in the evening sample.

EFFECTS OF COLD WEATHER. On eight occasions inspectors visited farms during spells of cold weather. The average results, obtained for milks taken on these occasions, were—fat 3.9 %, solids-not-fat 8.7 %. After one terribly cold, sleety night the morning milk contained 4.1 % of fat, and 9.0 % of solids-not-fat. On another occasion when the River Avon froze in the night, two samples of morning milk had 3.8 % and 4.4 % of fat respectively, and 8.8 % and 9.5 % of solids-not-fat, respectively. These figures do not give any support to the theory that low quality is due to cold weather.

EXCESSIVE DRINKING BY COWS. This has been given by a farmer as an explanation of the excess water in his milk (*B.F.J.*, 1921, 18). On this subject experiments were made by the direction of the Board of Agriculture (Intelligence Division Report for 1910, p. 8): "For four weeks the milk of seven cows was regularly analysed. Part of the time salt was given with the food, and the times of drinking were varied according to a definite plan. The result of the experiment pointed to the following conclusions: "(1) That periodical doses of common salt administered to cows, even to the extent of purging them, does not necessarily cause them to consume excessive quantities of water. (2) That the amount

of water consumed by cows has no direct bearing on the composition of their yield."

When a farmer was prosecuted at York for milk adulterated with 10·5 % of water, he stated that it was chiefly from one cow, which was newly calved, and which had drunk about 20 gallons of water. It was a second offence, and he was fined £15 (*B.F.J.*, 1929, 58).

The summer of 1911 was an unusually dry one, and comparisons were made between the rainfall and the average composition of Birmingham milk for ten years, to see if there was any relation. Comparisons are given below between months of August and September in 1902–1911, which had highest and lowest rainfall.

RAINFALL AND COMPOSITION OF MILK

MONTH.	Rainfall, inches.	Solids-not- fat %.	Fat %.	No. of months in average.
August . . .	4·9–5·2	8·65	3·6	2
„ . . .	0·9–1·4	8·65	3·55	2
September . .	2·6	8·65	3·7	2
„ . . .	0·9–1·0	8·7	3·73	3

It is evident that the composition of the milk kept constant whatever was the rainfall. Milks containing less than 8·0 % of solids-not-fat were not included in the calculations, being adulterated.

CONCLUSIONS AS TO LIMITS. In the writer's opinion, milks containing 8·3–8·5 % of solids-not-fat may be genuine, and legal action is not advisable unless the milk taken at the farm is of good quality, in which case a higher standard than 8·5 % may be used for the calculation. With regard to fat, it should be remembered that the limit of 3·0 % is a low one, being about 0·6 % below the average for genuine milks. On the other hand, a low fat is less likely to be due to fraud than low solids-not-fat. It may be due to an excessive interval between milkings, to accidental loss of cream, to improper distribution of the strippings, to carelessness in mixing before filling up one churn from another, or to the presence of fore milk. The variation in fat from day to day, also, is greater than that in solids-not-fat, and there is the added difficulty of the identification of the meal as evening or morning, as the amount of fat in the one cannot be calculated from that of the other and the churns may be incorrectly labelled, as to meal, by the farmer.

When cows yield milk below the limits, the farmer should be asked to improve the feeding of his cows, or to alter the intervals, so that the cows shall produce merchantable milk.

A number of statistics have been published showing what percentage of milk from herds of cows were below the presumptive limits. These figures are not very helpful unless they indicate *how much* the milks were below the limits. Obviously 22·4 % of milks

containing 8·4 % of solids-not-fat suggests a very different conclusion to 22·4 % of milks containing 8·1 % of solids-not-fat, but both may be included as being under 8·5 %.

In the table given on a previous page (197) 14 % of the farm samples contained 8·3 % and 8·4 % of solids-not-fat, and only 6 % were below 8·3 %; while only 3 % of the milks contained less than 3·0 % of fat. The conclusion drawn from twenty-one years' experience of visits to farms is that milks sent by farmers to Birmingham, which are low in solids-not-fat, are usually defective owing to adulteration, and not to the cows yielding poor milk. In the comparatively few cases in which the cows yielded poor milk, the cause was to be found in defective feeding, particularly in war-time, in some cases combined with abnormal intervals between the times of milking.

PROBABILITY OF MIXED MILKS BELOW LIMITS BEING GENUINE. The following table is calculated from figures given by Richmond ("Dairy Chemistry"; see also *S.P.A.*, 1917, **42**, 123):—

ODDS AGAINST MIXED MILK BEING GENUINE

FAT :

Below 3·0 %	137 to 1
„ 2·9 %	276 to 1
„ 2·8 %	664 to 1
„ 2·7 %	1,510 to 1
„ 2·6 %	3,450 to 1
„ 2·5 %	7,700 to 1

SOLIDS-NOT-FAT :

Below 8·5 %	46 to 1
„ 8·4 %	333 to 1
„ 8·3 %	1,690 to 1
„ 8·2 %	3,130 to 1
„ 8·1 %	10,000 to 1
„ 8·0 %	50,000 to 1

Tocher, in his monograph on Scotch cows (*op. cit.*, pp. 111, 113), lays great emphasis on the number of cows yielding milk of a particular quality, and gives the following figures in relation to total solids :—

ODDS AGAINST THE MIXED MILK FROM THE STATED NUMBER OF COWS BEING BELOW THE FOLLOWING PERCENTAGES OF TOTAL SOLIDS

No. of Cows.	TOTAL SOLIDS.		
	Below 12 %	Below 11·75 %	Below 11·5 %
5 . . .	35 to 1	200 to 1	2,000 to 1
10 . . .	177 to 1	2,229 to 1	48,543 to 1
15 . . .	1,054 to 1	42,016 to 1	4 millions to 1
20 . . .	5,969 to 1	769,230 to 1	333 millions to 1

As the total solids are made up of the solids-not-fat and fat, it will be seen from the above table that the odds against the mixed milk of fifteen Scotch cows being below the minimum presumptive limits of 8.5 % and 3.0 %, making 11.5 % of total solids, is four millions to one.

ADULTERATION

The prevention of milk adulteration is probably more difficult than that of any other food. The number of people who deal in milk is very large, and its adulteration requires no skill; also care in mixing before selling is necessary. Its sale, also, is rapid. A retail dealer may sell his watered milk within two hours, and a farmer's adulterated milk may soon be mixed with other milk for pasteurising. In each case the interval of time in which samples can be taken is short.

Before the issue of the Sale of Milk Regulations, 1901, milk was in the same position as most other foods, and in the absence of any legal standard or limit, each analyst had the obligation of deciding whether any particular sample of milk was genuine or adulterated. The Sale of Food and Drugs Act, 1899, gave the Board of Agriculture power to make "Regulations for determining what deficiency in any of the normal constituents of genuine milk . . . shall . . . raise the presumption until the contrary is proved, that the milk . . . is not genuine, . . . and the analyst shall have regard to such Regulations."

The Sale of Milk Regulations of 1901 fixed 3.0 % of milk fat and 8.5 % of milk solids other than milk fat, as presumptive limits. It is incorrect to describe these figures as standards or absolute limits.

References to appeal cases which have decided that the uncontradicted certificate of a public analyst is sufficient evidence to "raise the presumption" of adulteration, have been previously given (p. 52).

The appeal case *Hunt v. Richardson* (1916) practically superseded the Milk Regulations (see p. 40) by the "appeal to the cow" (see p. 197), and was followed by *Grigg v. Smith*, (see p. 222).

At times vendors of adulterated milk have escaped punishment by telling the inspector that the article was "milk and water." This defence is now prevented by sect. 4 of the 1922 Act. The same section prohibits the addition of colouring matter, dried milk, etc. (p. 53). The legal requirements as to labelling separated milk containers, and the exhibition of the vendor's name and address, have previously been mentioned (p. 53).

TONING. At the time of the issue of the Milk Regulations, 1901, it was suggested that the effect of them would be extensive reduction of good quality milk to the legal limits. To see if such

"toning" had happened in Birmingham, a comparison was made between the three years before the issue of them and the three years after. In the earlier period 68.9 % of the samples contained 12.0 % of total solids or more, and in the later period the proportion was practically identical, 69.0 %. A comparison was also made of the ratio between those with 11.0–11.4 %, and those 11.5–11.9 %. In the first period it was as 10 to 22, and in the second period the proportion of the better milks had increased, the ratio being 10 to 30. "Toning," therefore, if done, was not enough to affect the figures adversely.

RAIN. Occasionally a defendant pleads that the excess of water in his milk is due to rain; one, for instance, stated that his churn of milk had been open all one night in a heavy downfall of rain. The inspector, on the assumption that the mouth of the churn was 14 inches, calculated that the actual rainfall would account for 13 oz. of water, while 83 oz. were actually present! The fine was £5 (*F. & S.*, 1897, Dec. 18). In another case, only 0.4 % of water could be due to rain, while 9 % was found. In this case the fine was £50 (Ministry of Health Report, 1923).

A sample of milk taken from a Birmingham vendor who had been serving two and a half hours in the rain was of good quality, and another vendor warned the inspector that any water would be due to rain, but no excess was found.

ISING OF CREAM. The rate at which cream rises depends on a number of circumstances:—(1) Temperature, the cream rises more quickly in a cooled milk. (2) Agitation, as in a cart, delays its rising, but it is not safe to trust to mixing in this way. (3) Previous treatment. Sterilised milk throws up its cream slowly, and the quantity is decreased; pasteurised milk also yields a smaller quantity. (4) The age of the milk, and also whether it has previously risen and been remixed are other probable factors.

Stock (*S.P.A.*, 1930, **55**, 535) has recently made a number of experiments. He found much less difference when milk was poured than when it was dipped or taken from a tap. See also Arnaud (*Analyst*, 1926, **51**, 406), Elsdon and Stubbs (*S.P.A.*, 1930, **55**, 124). Some separation of cream is probable in fifteen to thirty minutes.

Legal decisions on the question have been given previously (p. 52). The sale of milk deficient in fat through standing is due to negligence, and is illegal.

DISTRIBUTORS. Milk dealers may be divided into three classes—producers, distributors, and retailers; and an attempt has been made below to show the different amounts of adulteration in the samples of milk sold by the three classes.

Samples from producers are taken at railway stations, milk depôts and a few from carts. For convenience this class has been called "farmers." Milk sold in shops is chiefly obtained from

wholesale dealers. Other samples taken at the stations are of milk which has been collected by a creamery from neighbouring farmers. The samples taken in the streets are in many cases milk which has been bought wholesale and in some cases contract milk supplied to institutions. These samples are described as "carts and creameries."

It is obvious that the farmer alone is responsible for adulterated milk taken from his churns and that samples sold in shops may have been previously adulterated, not by the retailer, but by the wholesale dealer or farmer. In the case of adulterated milk from shops an attempt is made to detect the real culprit. The following is an interesting series of samples :—

MILK FROM COWS TO SHOP

	Taken from :	Shop.	Wholesaler's cart.	Railway station.	Farm.
Fat %	.	2.30	2.15	3.60	4.15
Solids-not-fat %	.	8.15	8.35	7.90	8.80

The similarity between the first two samples showed that the adulteration had not been done by the shopkeeper. The differences between the samples taken from the cart and the station indicated the addition of separated milk by the wholesaler (who afterwards admitted the purchase of separated milk), while the differences between the farm milk and that from the station proved the addition of water at the farm. The farmer was fined £5, and the wholesale dealer £20.

The following table gives comparative figures for adulteration found in milk supplied by the various classes of vendors :—

MILK FROM FARMERS AND DISTRIBUTORS

PERCENTAGE OF ADULTERATION

	1889—	1894—	1904—	1914—	1924—9
Farmers	14.1	12.0	10.4	9.0	7.7
Carts, creameries, shops	23.4	15.0	8.9	7.3	3.3

COMPARATIVE ADULTERATION FIGURE

Farmers	—	—	3.7	3.4	3.3
Carts, creameries	—	—	6.5	4.0	2.0
Shops	—	—	7.2	7.0	4.6
Bottled milk	—	—	—	—	1.4

The improvement in the percentage of adulteration in the forty-one years is notable, that of farmers' samples falling from 14.1 % to 7.7 % ; the fall in the other samples is much more marked, 23.4 % to 3.3 %. In the last period it was less than half the farmers' class, while in the first it was considerably greater. These figures, however, must not be pressed, as in recent years more samples have been taken from each adulterating farmer.

The comparative adulteration figures (p. 114) are much less affected by sampling, and it will be seen that in the twenty-six years

available the improvement in the farmers' samples has been much less than in the other two classes. In comparing the three classes for a particular period, it must be remembered that the adulteration of samples sold at shops may have been previously done by the farmer or wholesaler, and that more care in mixing is necessary during retail sale in small quantities.

During recent years there has been an increasing tendency by Birmingham wholesalers to have their milk brought by motor lorries to their depôts, instead of by railway. A few years ago farmers thought that road-borne milk did not run the risks of sampling to which rail-borne milk was liable.

For the years 1926-9 comparisons have been made between these two classes. The rail milk showed 5·7 % of adulteration, and the road 9·7 %; the comparative adulteration figures were 2·8 and 4·5 respectively. It is obviously very necessary to sample milk brought into depôts.

PERCENTAGES, RELATION TO POPULATION, FINES. Below are given adulteration figures for Birmingham, based on a limit of 11·5 % of total solids, and which do not include any samples adulterated with preservatives only. The figures for England and Wales and London are based on official reports, and will include a small proportion of samples adulterated with preservatives only :—

PERCENTAGE OF ADULTERATION, NUMBER OF SAMPLES AND FINES

	1873-	1870-	1889-	1899-	1909-	1919-	1929	1930
England and Wales,								
Adulteration %	—	16·6	11·9	11·0	10·8*	8·3	7·8	6·6
London „	—	22·6	19·4	12·7	9·5*	3·9†	3·5	2·7
Birmingham „	53·2	27·5	16·6	10·1	9·4	6·4	8·7	5·0
Annual samples								
per 100,000								
persons .	9	35	70	119	210	259	257	245
Average fine per								
vendor fined .	15s.	£1 6s.	£1 10s.	£3 3s.	£7 18s.	£17 4s.	£27 1s.	£5 0s.

* 1909-13 only.

† 1920-8 only.

In the first period more than half of the samples of milk bought in Birmingham were adulterated and in the next period more than a quarter of them, and one is not surprised that the 1881 Report of the Local Government Board remarked: "Birmingham still maintains the distinction, which it has for some years enjoyed, of having a larger proportion of its milk adulterated than any other great town, in the kingdom."

In 1889-98, however, the figure for Birmingham was lower than that for London and for the next two decades about 1 % lower than England and Wales as a whole and about 2 % in 1919-28, but in 1929 Birmingham showed an increase of 2·3 %, followed by a fall to 5·0 % in 1930. In recent years London has shown very low adulteration figures.

PROFITS OF ADULTERATION. The importance of the adulteration of milk to the community may be illustrated by some Birmingham calculations. In 1910 the amount of "milk" sent by thirteen farmers to Birmingham was 400 gallons per day; of this 42 gallons was water. It may be noted that the addition of 10 % of water enables a farmer to keep ten cows instead of eleven. In 1914 a farmer was receiving 73s. per week for water, and in 1918 the illegitimate profit of another farmer was £1 per day. In 1929 two farmers were *daily* sending about 27 gallons of water as milk.

On the assumption that $\frac{1}{4}$ pint of milk is taken daily by each inhabitant of Birmingham, the annual milk bill is about £1,000,000. After allowing for milk that is naturally poor, it is probable that 2 gallons out of each 1,000 is water, which corresponds to about £2,000 per annum for water. This is not satisfactory, but if milk adulteration had been at the same rate as fifty years previously, about £110,000 would at the present time have been annually paid for water. Sometimes milkmen's bills may be an additional water rate.

There is another point of view. Some vendors appear to consider adulteration fines a part of ordinary working expenses—unfortunate, no doubt, but easily paid out of the profits. Here is one farmer's record:—1910, fines £10; 1911, £10; in 1914 one sample had 7 % added water; on the second day, the milk was sent to the country station, but the farmer fetched it away *after the arrival of the post*. He was called up and escaped with 1 guinea costs. While he was on war service the milk from his farm was genuine. In 1915 he was fined £50; in 1918, £160; and in 1919, £300. He appealed to Quarter Sessions, and with very good luck; the conviction was quashed. Subsequent samples have been genuine.

A retail dealer with a milk round was not much better. In 1906 he was fined £1, and absconded when another adulterated sample was bought; in 1909 he was fined in Middlesex. In 1911 he was fined £5 at Coventry; in 1914 he was fined £10 in Birmingham; and in the next year, when he was selling more than 150 gallons a day, sent to prison for three months with hard labour for selling milk with 23 % of added water. In 1918, after selling three samples adulterated with 18–28 % of water, he absconded to London. His further history is unknown.

A Sheffield milk dealer who had just been fined, boasted that, as he knew he would be safe from further sampling till his case had been heard, he had continued his "mixing" and had made more in the meantime than the amount of his fine and costs (*F. & S.*, 1893, April 22).

These cases emphasise the fact that small fines, and even moderate fines, may be useless, and that penalties must be severe enough to make adulteration unprofitable.

PROSECUTIONS FOR ADDED WATER. *Sproatley.* Extraneous water 3·6 %, containing living animalculæ. The water from the pump was similar and swarming with organisms dangerous to health. Fines and costs £44 5s. (*B.F.J.*, 1910, 115).

Ramsgate. Extraneous water 10 %, and deficient of 10 % of fatty solids. A lump of ice had been placed in the bowl to keep it cool. Fine £5 (*B.F.J.*, 1913, 195).

Birmingham. Added water 60 %. Fine £50 and 5 guineas costs. (The vendor had been previously fined £10.) (1918 Report.)

Birmingham. Solids-not-fat deficient in nine samples $5\frac{1}{2}$ –8 % ; fat deficient in two other samples, 20 % and 23 % respectively. Fines £95. (The vendor had been fined £40 in the previous year.) (1918 Report.)

Rugby. Extraneous water 6 % when calculated on 9·1 % of solids-not-fat. Milk in the serving bucket contained 8·56 % of solids-not-fat, and that in a churn on the cart 9·1 %. The defendant attributed the excess of water to snow having fallen into his serving bucket. Fine £5 (Warwickshire County Report, 1917).

Bootle. Solids-not-fats 7·88 %. The deficiency was attributed to a lime deficiency in the soil, and the case was dismissed (*B.F.J.*, 1928, 68).

Birmingham. Solids-not-fat 8·22 % deficient in seventeen samples, and fat 13 % and 20 % deficient in two others. Samples taken at the farm were of very good quality, and taking them as a standard, it was calculated that 14 of the 95 gallons sent on the Sunday were water, and 13 of the 92 gallons sent the next day. At that rate about 100 gallons of water would be sold as milk in a week. The samples were taken from a lorry at a milk depôt. Fine £100 (*B.F.J.*, 1929, 90).

Stowmarket. Added water not less than $8\frac{1}{4}$ %. The inspector admitted that “added” did not necessarily mean that water had been put in. Fine 10s. (*B.F.J.*, 1930, 48).

Rugby. Added water 27 % and 18·5 % respectively. The vendor's boy admitted adding a bucket of water to each churn. Fine £3 in each case (*B.F.J.*, 1930, 50).

Williton. Added water 11 %. The defence objected to the word “added” and suggested the water was natural, and that it could not be proved to have been poured in. Also, that the fat being above the average disposed of the theory that extraneous water had been introduced. Case dismissed (*B.F.J.*, 1930, 59).

Poole. Added water 2 %, and fatty solids 2 % deficient. The vendor, who had previously been prosecuted three times, was fined £2 (*B.F.J.*, 1930, 88).

Boston. Added water 26·47 %, and deficient in fat 32 %. The farmer stated that he suspected his servant, because he saw sand in the milk, and that he had watched him adulterate the milk. The

servant was condemned to two months' imprisonment (*B.F.J.*, 1930, 85).

Yeovil. Actually added water 12 %, an opinion based on 7.96 % of solids-not-fat, and a freezing-point of minus 0.485°, instead of 8.5 %, and minus 0.55°, respectively. The sample taken at the farm was of good quality. Fine £2 (*B.F.J.*, 1931, 18).

Abercynon. Extraneous water 60 %, and deficient in fatty solids 69 %. It was stated that the mixture was intended to be added in small quantities to other milk. Fine £20 (*B.F.J.*, 1931, 68).

Birmingham. Added water 9 % and 31 %, respectively. The freezing-point of the first sample was minus 0.496 %, and of the second 0.413°, instead of minus 0.530°. The freezing-point of milk taken at the farm was minus 0.542°. Fine £10 in each case (*B.F.J.*, 1931, 89).

Worcester. Deficient in fatty solids 16.6 %, and in non-fatty solids 18.8 %. On the visit to the farm a sample taken from the 10½ gallons produced was found to contain 10 % of water. The churn, however, was left unwatched while the officers watched the milking. On a second visit only 9 gallons was obtained from the cows, and the quality of it was good. Fine £10 (*B.F.J.*, 1931, 88).

Tunbridge Wells. Extraneous water 2 %. The defendants proved a warranty, but it was not accepted as notice had not been given to the authority to take a sample of milk in course of transit or delivery. Fine 3 guineas (*Grocer*, 1931, Nov. 28).

Reigate. Selling milk to which water had been added, contrary to sect. 4 (1) of the Milk and Dairies (Amendment) Act, 1922. The samples contained not less than 7 %, and 9 %, respectively, of added water ; which opinion was confirmed by their freezing points. Fine £2. (*B.F.J.*, 1932, 18 ; *Analyst*, 1932, 57, 164).

PROSECUTIONS FOR DEFICIENCY OF FAT. *Margate*. Appeal to Quarter Sessions against a conviction for milk "from which a part had been abstracted, to wit, 19 % of its fat or cream." The appeal was allowed, the Recorder holding that the cream had not been physically abstracted, though skimmed milk or water might have been added (*B.F.J.*, 1902, 247).

Birmingham. Fat deficient 40 %, 16 %, and 13 %, in samples taken from three tins on a milk cart. Genuine separated milk was also carried for mixing. Each of two brothers was fined £10 (1906 Report).

North London. Fatty solids 7 % deficient. Defendant admitted that for over three years he had been adding 5 to 6 pints of separated milk to each churn of whole milk. Fine £50 (*B.F.J.*, 1911, 156).

Marylebone. Fatty solids deficient 26 %. It had been taken from a penny-in-the-slot machine, and the vendor stated that he had stirred the milk each hour. Fine and costs 33s. (*B.F.J.*, 1912, 236).

A vendor cried "Pure milk 1½d." His barrow was marked "Pure skim milk," but a coat covered the word "skim." Fine £15 (L.G.B. Report, 1912).

Lancaster. Appeal to Quarter Sessions against a conviction for selling "milk deprived of 7 % of fat." Evidence was given that the milk had been taken, after an interval of fifteen hours, from a herd which included some newly calved cows. The chairman suggested that the use of the word "deficient" would have been more fair than "deprived." The appeal was dismissed, but the fine reduced to 5s. (*B.F.J.*, 1913, 5).

Newmarket. Fat deficient 40 %. The defendant had a separator at home, and later samples were genuine. Fine £3 (*B.F.J.*, 1929, 108).

West Ham. Fat deficient 21·7 %. The milk from the cows in poor condition was sold in the borough, and the better milk sent away to wholesalers. Fine £20 (*B.F.J.*, 1929, 97).

Hamilton. Fat deficient 12 %. The sample was taken from a can, containing about ½ gallon, from which about 2½ gallons had been sold retail. The Sheriff fined the respondent £3 as she had not shown that the deficiency could not have been prevented (*B.F.J.*, 1930, 6).

Chipping Sodbury. Fat deficient 25·7 %. The sample was taken from the milk of one cow, which was said to be in an excited condition when milked. Three days later the milk was satisfactory. Fine and costs 6½ guineas (*B.F.J.*, 1930, 49).

Leeds. Grade A milk seriously deficient in fat. The defendant pleaded warranty, and complained that he could not give notice within sixty hours to have his farmer sampled as he did not know for seven days that the milk was adulterated. Paid costs (*B.F.J.*, 1930, 87).

Croydon. Bottled milk containing only 1·88 % of fat. Evidence was given that the roundsman had adulterated the milk and he was fined £5. His employers were considered to have been careless, and were fined £2 (*B.F.J.*, 1931, 67).

Birkenhead. Fat deficient 13%. The sample was taken on a Wednesday, and milk taken from the same cows on Sunday was genuine. The case was dismissed, the chairman saying that the Corporation did not take immediate steps to obtain the second sample (*B.F.J.*, 1931, 99).

PROSECUTIONS FOR STARCH, SALT, ETC. *Glasgow.* Water 22 %, common salt 0·21 %. Fine £20 (*Analyst*, 1886, 11, 238).

Dublin. Water 16 %, starch 2 %. The Government analysts did not find added water, and only 0·17 % of starch. Fine £3 (the Public Analyst in a subsequent letter suggested that, during the forty-seven days that had elapsed before the Government analysts received the sample, the starch had been converted into

dextrin and glucose, which were included in the solids-not-fat and masked the presence of added water) (*F. & S.*, 1896, Aug. 29).

London, West Ham. Added water 44 %, fat deficient 25 %, and an excess of chlorine equivalent to 180 grains per gallon of common salt. The vendor said that he was selling it as "milk and water." Fine £5 (*B.F.J.*, 1899, 155).

Wigan. Extraneous water 5 %, wheat flour 0.2 %, and egg in the proportion of the white of one egg to two quarts of milk. There was a second similar offence. The addition of eggs was admitted, but that of flour denied. Fine £10 (*B.F.J.*, 1903, 41).

Birmingham. Saltpetre 5 parts per 100,000. It was said that the addition was to remove the flavour of turnips. Paid costs (*B.F.J.*, 1913, 33).

Edinburgh. Extraneous water 9 %, cane sugar 0.36 %, common salt 0.32 %. Fine £10 (*B.F.J.*, 1916, 291).

Brentford. Copper sulphate 14.5 grains in the sample, and deficient in fatty solids. Fine £2 (*B.F.J.*, 1926, 109).

Highgate. Fatty solids deficient 70 %, and composed of a mixture of starch and water. The vendor had been calling out "Lovely Milk." Fine £20 (*B.F.J.*, 1920, 120).

PROSECUTIONS FOR VARIOUS OFFENCES. *Birmingham.* Vendor fined £10 for obstruction. On learning the sample was to be analysed, she snatched it and poured the milk back. Another sample contained 6 % of water in excess. The vendor's husband had been previously fined for adulteration (1900 Report).

Birmingham. Vendor was fined 10s. for not having his name and address on his milk can (1902 Report).

Marylebone. Extraneous water 16 %. The defendant stated that as milk was scarce he had added the contents of tins of condensed skim milk, and also the washings of the tins. The sample had been taken on Easter Monday. Fine £3 (*B.F.J.*, 1906, 140).

London, Marlborough Street. The defendants were fined £2 for carrying on the trade of milk purveyors without being registered in the district (*B.F.J.*, 1909, 211).

Birmingham County Court. A milk dealer obtained £15 for breach of contract from a farmer who had supplied him with adulterated milk. The farmer had been fined, but the dealer had covered himself with a warranty (*B.F.J.*, 1909, 230).

Evesham. Cowman prosecuted for doing wilful damage to his master's milk to the extent of 45s. He had been seen to take water from a dirty rain tub. On three successive mornings about 20 % of water was present in the milk sent to Birmingham, but on the fourth morning the quality was very good. He admitted adding 8 quarts of water, and was sent to prison for two months' hard labour (1916 Birmingham Report).*

* See Addenda, p. 577.

West London. Extraneous water 50 %. The vendor said it was "new milk and water," although the churn, from which he was serving customers, was marked "Pure milk." The magistrate dismissed the summons, thinking the case was not made out; as there was no evidence that it was not milk and water, the proportion did not matter (*B.F.J.*, 1916, 313).

Neath. Extraneous water 18 %. The vendor owned a creamery, and if his farmers did not supply a sufficient quantity of milk the balance was made up by the addition of a reconstituted milk made from milk powder and water. Fine £10 (*B.F.J.*, 1916, 313).

West London. Knowingly selling milk to which water had been added contrary to the Milk (Amendment) Order, 1917. The two samples contained separated milk and 27 % added water. The vendor was sentenced to six weeks' hard labour (*B.F.J.*, 1918, 34).

Birmingham. Giving a false warranty with milk which contained 18 % of added water. Fine £20, and also £30 for a similar offence to another customer with milk containing 8 % of added water (1924 Report).

Sittingbourne. In a fog a dairyman was seen to add a pellet containing rennet to a rival dairyman's milk. He was fined £5 for damaging the milk (*B.F.J.*, 1922, 26).

Smethwick. Extraneous water 11 %, and fatty solids deficient 20 %. The vendor admitted that the sample had been made from dried milk (*B.F.J.*, 1924, 17).

Kensington. Failing to keep clean the interior of a cart, and vessels used for milk; being unregistered, and obstructing the inspector. The defendants were fined £50 (*B.F.J.*, 1932, 17).

ANALYSIS

TOTAL SOLIDS. (1) *Approximate Determination.* Pipette 5 gm. into a porcelain dish $2\frac{3}{4}$ inches in diameter, and add 2 drops of methylated spirit containing 10 v/v of 10E.HÅ. Evaporate to dryness and dry in water oven two and a half to three hours. The pipette should deliver 4.82 gm. of water in five seconds, touching the surface of the water at the end. It will then deliver a close approximation to 5 gm. of average milk by draining about eight seconds. Such dishes only alter very slowly in weight, and need only be weighed occasionally. Time is also saved by having, for each dish, a small numbered metal box, containing lead shot, to be used as a tare. The weight of the milk solids is then obtained without subtracting the weight of the dish; the latter should, however, be approximately known in case of an accident to the tared box. The dishes should have flat bottoms.

(2) *Accurate Determination.* Deliver about 5 gm. from a pipette, drain one minute into 3-inch flat-bottom metal dish, blow out, and

weigh. (This weight is also taken as that for Adams' fat determination.) Evaporate, etc., as above, but without acid.

FAT. Adams' Method. Deliver about 5 gm. on to a coil as evenly as possible, draining about one minute, as in the above determination of total solids. Leave overnight, and complete drying by passing quickly through a flame. Extract three hours with ether (sp. gr. 0.720) in a 30-ml. Soxhlet extractor, distil off the ether, dry in water oven one and a half hours, and a further half hour if necessary. If there be any indication of water, a little absolute alcohol and ether are added before drying. The coils are $22 \times 2\frac{1}{2}$ inches and should weigh about 6 gm. Although the coils are nominally fat-free, it is advisable to extract for three hours with ether before use; sixteen may be done at once in a large Soxhlet. Two or three of these coils are again extracted, and the average amount obtained is used as a correction for each determination. The ether used should be dried previously with calcium chloride, and redistilled.

Gerber Method. (See Richmond, *S.P.A.*, 1905, **30**, 77, 326; 1918, **43**, 405; Day and Grimes, *S.P.A.*, 1918, **43**, 123, 215; Harvey and Harvey, *S.P.A.*, 1923, **48**, 213.) Deliver 10 ml. of the sulphuric acid from the automatic pipette into butyrometer, and also 1 ml. of amyl alcohol. Deliver 11 ml. (11.23 gm.) of milk, running it down the side to avoid mixing. Touch the side of the tube with the pipette, but do not blow out. Shake till uniform, inverting periodically to allow the acid from the stem to mix. In cold weather it is advisable, when a number of samples are being determined, to lay the tubes on wood after shaking, and put them all into the centrifuge at once. Whirl at 800–1,000 revolutions per minute, put in water at 65° C. and read after about five minutes. Sterilised milks should be whirled a second time after reading, and perhaps a third time, to make sure all the fat has separated. If formic aldehyde be present, the liquid will be violet when the amount of fat is read. A violet colour while shaking may not be due to that preservative. A golden-brown colour may indicate nitrates or nitrites, and the tube may burst (Stokes, *S.P.A.*, 1897, **22**, 321).

The apparatus and chemicals should be tested for correctness. The pipette should deliver 11.02 gm. of water at 15.5° C. in five seconds, touching the water at the end. Eight small divisions of the butyrometer (0.8 % of fat) should equal 0.1 ml., and from the bottom of the scale to the inner end of the neck should be 20.5–21.5 ml. The amyl alcohol should have sp. gr. 0.814–0.815, and B.P. 130–132° C. There must be no oily residue when 2 ml. are added to a cooled mixture of 10 ml. of acid and 10 ml. of water and centrifuged. The sulphuric acid should have sp. gr. 1.820–1.825. It is prepared by pouring commercial sulphuric acid into 250 ml. of water in a beaker, and cooling. The mixture is returned to the

W.Q. and allowed to stand overnight to cool. The sp. gr. is taken with a hydrometer ($1^{\circ}\text{C.} = 0.001$ sp. gr.). The following table shows how much water containing 5 ml. of Liq. Ferri Persulph. (B.P.) should be mixed with 10 lb. (practically a W.Q.) of acid for each sp. gr. :—

1.825	.	0 ml.	1.830	.	58 ml.	1.835	.	128 ml.
6	.	12 „	1	.	71 „	6	.	144 „
7	.	25 „	2	.	84 „	7	.	160 „
8	.	35 „	3	.	98 „	8	.	180 „
9	.	45 „	4	.	113 „	9	.	200 „

Gottlieb Method. (See Weibull, *Analyst*, 1898, **23**, 259; Popp, *Analyst*, 1904, **29**, 112; Richmond, *S.P.A.*, 1906, **31**, 218.) Deliver about 5 gm., draining one minute, as for total solids, into a Werner-Schmid tube. Add 0.5 ml. 6E.AmOH, then 5 ml. methylated spirit, then 12.5 ml. ether, and finally 12.5 ml. B.P. petroleum spirit, shaking after each addition. If the tube is slightly warmed by the hand before corking, liquid will not be blown out when the cork is removed. Insert into the neck a cork bearing tubes similar to those of a wash bottle, but having the lower end turned up. Blow off as much of the ethereal layer as is possible into a flask, and further extract with two quantities of 15 ml. of a mixture of equal parts of ether and petroleum spirit. (The recovered solvent will serve.) Distil off the solvent and dry to constant weight. Wash flask with petroleum spirit, dry, weigh flask and residue, and subtract from total weight. With sour milks it may be necessary, after adding the methylated spirit, to stand the tube in warm water for a few minutes, and shake to break down any lumps. A separator may be used instead of the Werner-Schmid tube.

Werner-Schmid Method. (See Stokes, *S.P.A.*, 1889, **14**, 29; T. E. Hill, *S.P.A.*, 1891, **16**, 67.) Deliver two quantities of about 5 gm. as for total solids into a Werner-Schmid tube, add 10 ml. HCl and boil with shaking till dark brown, but not more than two minutes. Cool, add 30 ml. ether, shake round, let stand ten minutes, blow off the ethereal layer as in the Gottlieb method, and repeat the extraction twice. Distil off ether, dry and weigh. About 5 gm. of sour milk should be weighed into the tube and diluted with an equal quantity of water, before the addition of the acid. Repeated extraction with ether is inadvisable, owing to the solubility of lactic acid in ether. A modification for malted milk has been given by Rose (*Analyst*, 1927, **52**, 92).

MILK SUGAR. (See Vieth, *S.P.A.*, 1888, **13**, 63; Richmond and Boseley, *S.P.A.*, 1897, **22**, 98.) Add 3 ml. mercuric nitrate solution to 100 ml. milk, mix by pouring between two beakers, filter and polarise in 200 mm. tube.

$$\text{Anhydrous lactose, \%} = \frac{100a - (1.075 \times F \times S)}{110.6 S}, \text{ where } a =$$

degrees of rotation, $F = \% \text{ fat}$, and $S = \text{sp. gr. of milk}$. The reagent is made by dissolving 5 ml. mercury in 96 ml. HNO_3 and diluting the product with an equal volume of water. See also Baker and Hulton (*S.P.A.*, 1910, **35**, 512), Richmond (*S.P.A.*, 1910, **35**, 516), and Bacharach (*S.P.A.*, 1923, **48**, 521). Davies has shown that the determination of chlorine may be applied to the approximate determination of lactose (*S.P.A.*, 1932, **57**, 83).

SPECIFIC GRAVITY. This should be taken with a delicate lactometer, and corrected to 60° F . A table for this correction is given in the Appendix (p. 559). The sp. gr. of milk taken an hour after milking is about 0.001 less than the true sp. gr. (Rachnagel's phenomenon, *S.P.A.*, 1895, **20**, 1).

FREEZING POINT. Last year (1931) the freezing-point method for detecting the dilution of milk has come to the fore, and has been mentioned in several prosecutions. During 1930 G. D. Elsdon applied the test to a number of samples which had low solids-not-fat. He reported: "In every case the information obtained by the application of the test was identical with that obtained from comparison with the corresponding 'Appeal-to-cow' sample. It is submitted that the freezing-point test is of conclusive value in the detection of added water in milk in all cases where corresponding 'Appeal-to-cow' samples are available" (Lancashire County Analyst's Report, 1930). (See Addenda, p. 577.)

Monier-Williams made a report on the method to L.G.B. (Food Report No. 22, 1914). Other papers have been given by Joseph and Marton (*S.P.A.*, 1924, **49**, 420), Andrew (*S.P.A.*, 1929, **54**, 210), Parker and Spackman (*S.P.A.*, 1929, **54**, 217), van Raalte (*S.P.A.*, 1929, **54**, 266), Henderson (*Analyst*, 1929, **54**, 747). Details of the method, with experiments on the method and a bibliography, are given in papers by Elsdon and Stubbs (*S.P.A.*, 1930, **55**, 423, and *J.S.C.I.*, 1931, 135 T, and *B.F.J.*, 1931, 72, 84, 93, 103).

OTHER DETERMINATIONS. *Ash, Chlorine and Nitrogen* have been given previously (pp. 70, 73, 63). *Immersion Refractometer.* See Elsdon and Stubbs (*S.P.A.*, 1927, **52**, 193; 1929, **54**, 318; 1930, **55**, 618). *Aldehyde Figure*, for determination of proteins. See Richmond (*S.P.A.*, 1906, **31**, 224; 1909, **34**, 209; 1911, **36**, 9; 1913, **38**, 254). *Nitrates* have been added to milk as a preservative, and also to cover the taste when cows have been fed on turnips. When such milk decomposes, *nitrites* are formed; the latter have been added to prevent formic aldehyde being detected. Papers on these subjects have been given by A. W. Stokes (*S.P.A.*, 1897, **22**, 320), G. A. Stokes (*S.P.A.*, 1912, **37**, 178), Elsdon and Sutcliffe (*S.P.A.*, 1913, **38**, 450), Elsdon and Smith (*S.P.A.*, 1922, **47**, 18). Experiments showing that the milk of cows dosed with moderate amounts of nitrates does not contain nitrates have been given by Krause. Lerrigo (*S.P.A.*, 1930, **55**, 433) advocates testing for nitrates

as a routine test for the detection of added water. *Sucrose* has been detected in milk, but only when it can be tasted (Muter, *S.P.A.*, 1880, **5**, 39). Elsdon has given a method (*S.P.A.*, 1918, **43**, 292). Mattick (*S.P.A.*, 1930, **55**, 37) has shown that *phenols* may occur naturally in sterilised milk. The official bacteriological tests for graded milk are given : *Analyst*, 1929, **54**, 235, and *B.F.J.*, 1929, 33.

CALCULATION OF THE NORMAL RELATIONS OF CONSTITUENTS. Richmond found that the percentage of ash in milk was equal to $0.36\% + 0.11 \times \text{proteids } \%$ (*S.P.A.*, 1901, **26**, 313).

Vieth (*S.P.A.*, 1891, **16**, 206) stated, and Richmond (*S.P.A.*, 1899, **24**, 199) confirmed, that the relation in normal milk between ash, protein and sugar is 2 : 9 : 13.

There has been considerable investigation as to the effect of fat and solids-not-fat on the sp. gr. of milk. Tables have been given in the Appendix (p. 560) of calculations based on Richmond's formula (*S.P.A.*, 1894, **19**, 81). See also Leonard (*S.P.A.*, 1900, **25**, 67 ; 1901, **26**, 318) and Harris (*S.P.A.*, 1918, **43**, 263 ; 1919, **44**, 200, 314). Richmond has devised a dairy slide-rule (*S.P.A.*, 1920, **45**, 218).

CALCULATION OF MILK ADULTERATION. Reference to the forms of milk prosecution certificates, and to evidence on milk, has been made in previous chapters (pp. 39, 56).

Although by the Sale of Milk Regulations the "addition of water" or the "abstraction of fat" may be "presumed," the writer prefers not to use these phrases unless a standard farm sample has been obtained. In other cases :—

$$\begin{aligned}\% \text{ of solids-not-fat deficient} &= (8.5 - n) 11.8 = W \\ \% \text{ of fat deficient} &= (3.0 - f) 33.3 = A,\end{aligned}$$

where n and f are the percentages of solids-not-fat and fat, respectively, in an adulterated sample. Obviously, A might be due to addition of water, abstraction of fat, or both. If it be smaller, or little greater than W , it should not be mentioned on a certificate, as is done sometimes, or the vendor may think he is accused of skimming, when the deficiency is due to watering the milk. When A is notably greater than W , a statement should be made that part of the deficiency is due to water, or the offence is apparently magnified.

With a standard farm sample containing $N\%$ of solids-not-fat, and $F\%$ of fat :—

$$\begin{aligned}\% \text{ added water} &= \frac{(N - n) 100}{N} = W \\ \% \text{ fat abstracted} &= \frac{(F - f) 100}{F} = A,\end{aligned}$$

but in this case W must be subtracted from A .

The amount of added water in a churn of milk containing G gallons is conveniently calculated as follows :—

$$\text{Gallons of water} = \frac{(N - n) G}{N}.$$

The following calculation is based on the assumption that butter contains 11 % of water, and that the sp. gr. of milk is 1.03 :—

$$\text{Pounds of butter removed} = 0.116 G (F - f).$$

If there has been abstraction of fat as well as addition of water, a correction must be applied to the fat of the farm milk before the last calculation be applied.

$$\text{Fat in watered farm milk} = 100 n F/N.$$

An actual example will illustrate the working. Evening milk taken at the station measured 10 gallons, and $n = 7.7$ and $f = 3.1$, while milk taken by inspectors at the farm gave $N = 8.4$ and $F = 4.2$. Water = $\frac{(8.4 - 7.7) 10}{8.4} = 0.8$ gallon. Fat in watered farm milk = $100 \times 7.7 \times 4.2/8.4 = 3.8$. Butter removed = $1.16 (3.8 - 3.1) = 0.8$ lb. Evidence was given at the prosecution as follows : “ 9.2 gallons of evening milk taken at the farm, mixed with 0.8 gallon of water, would give 10 gallons of milk, containing 7.7 % of solids-not-fat, as is present in this sample, and 3.8 % of fat, but the removal of fat equal to 0.8 lb. of butter would be necessary to reduce the fat to 3.1 % as is present in this sample ” (cp. p. 56).

When an excessive amount of fat is present in a watered milk, allowance should be made for it in the calculation. For limit milk the formula of L. J. Harris (*S.P.A.*, 1919, **44**, 318) becomes

$$W = 100 - f - 11.4 n.$$

Leonard and Smith have shown (*S.P.A.*, 1896, **21**, 283) that the separation of fat makes no difference to the ratio between solids-not-fat and water of the milk. For a prosecution when 4.68 % of fat was present, see *B.F.J.*, 1929, 107, 120, 130 ; 1930, 10.

Richmond (“ Dairy Chemistry ”) has proposed the following formula, where $G = (1,000 \text{ sp. gr.}) - 1,000$:—

$$W = 100 - \frac{100 (G - f)}{34.5 \text{ or } 36},$$

where the former divisor represents the minimum, and the latter the probable amount.

Milks on souring do not lose nitrogen, and in such cases the minimum adulteration can be calculated on the assumption that milk contains 0.5 % of nitrogen (cp. Smetham and Ashworth, *S.P.A.*, 1897, **22**, 172).

Occasionally, owing to a vendor having been known to have bought separated milk, the composition of a sample expressed as

genuine milk, separated milk and water may be required. In the following formulæ the separated milk is assumed to contain 9.0 % of solids-not-fat, and 0.2 % of fat. For skimmed milk the corresponding figures are 8.7 % and 1.0 %, and these figures may be substituted :—

$$\text{Genuine milk \%} = \frac{100 (9f - 0.2n)}{9F - 0.2N}$$

$$\text{Separated milk \%} = \frac{100 (Fn - Nf)}{9F - 0.2N}$$

$$\text{Water \%} = 100 - \frac{(100Fn - 20) + f(9 - N)}{9F - 0.2N}.$$

In a prosecution, $f = 0.7$ %, $n = 5.9$ %, and the calculated constituents were 17 %, 49 %, and 34 %, respectively. For a graphical method of solution see Liverseege (*J.S.C.I.*, 1908, 604).

L. J. Harris has constructed a phase-diagram (*S.P.A.*, 1918, 43, 376), which may be used to indicate if a single figure postulates possible, or impossible, other figures for genuine milk, particularly if additional lines for solids-not-fat be ruled on it.

Freezing-point. In the absence of a sample from the farm, the following formula may be used for the calculation of the minimum percentage of added water, but one prefers to use the actual freezing-point of the farm sample instead of the -0.53° in the equation :—

$$\text{Water, \%} = \frac{(-0.53^{\circ} - \text{freezing-point of sample}) 100}{-0.53^{\circ}}.$$

FORE MILK

When a cow is partly milked, the "fore milk" first obtained contains less fat than the later part, or "strippings." Four cows were milked under the observation of a Birmingham inspector, fourteen and a half hours after the previous milking. The first part, 14 quarts, had 1.5 % of fat, and the subsequent 17 quarts, 3.7 %. For one cow, the two parts contained 1.0 % and 3.9 %. The greatest difference in solids-not-fat was 0.2 %.

One defendant, who admitted he knew nothing about milking cows, used to take milk from the cow, sell it, and then go back to the cow for some more ! He was fined £5 for the sale of milk deficient of 36 % of its fat (*B.F.J.*, 1922, 58).

The appeal case *Grigg v. Smith* (*Analyst*, 1917, 42, 323 ; *B.F.J.*, 1917, 141) arose from the sale of the fore milk, which contained only 2.6 % of fat, the remainder being left for the calf. Following *Hunt v. Richardson*, the conviction was confirmed, one of the judges remarked that a farmer is now entitled by law to give preference to his own calves over the babies of his customers. A farmer, who was

sending fore milk to Birmingham, told an inspector that "the calves thrive so much better" when they sucked the remaining milk.

The milk from one cow fed on poor pasture was 28 % deficient in fat. *Williams v. Rees* (1918) ruled that, though it was not merchantable, it was "as it came from the cow."

The above decision has usually been followed, but as the result of appeal cases on the rising of cream (p. 52) the Sheriff-substitute at Lanark fined a vendor for milk deficient in fat, which had been taken from the first part drawn from one cow. He was unable to see that leaving the richest of the milk in the udder of the cow should have any different effect from leaving it at the top of the churn (*B.F.J.*, 1927, 117 ; 1928, 17).

SEPARATED MILK. SKIMMED MILK

The Milk Regulations of 1901 gave for these a presumptive limit of 9.0 % of milk-solids. This proved unsatisfactory, as a somewhat diluted whole milk might comply with this requirement. The revised Regulation of 1912 substituted 8.7 % of solids-not-fat for the milk-solids limit.

The examination of samples of separated milk, passed as genuine, gave the following range of composition :—

COMPOSITION OF SEPARATED MILK (Forty-two samples)

Percentage of solids-not-fat .	8.6	8.7-	8.9-	9.1-	9.2-9.5	Total.
Percentage of samples .	5	31	29	26	9	100
Percentage of fat .	0.1-	0.3-	0.5	0.7-1.0	.	Total.
Percentage of samples .	31	50	12	7	.	100

If the samples containing 0.7-1.0 % of fat were separated milks the separator must have been working very badly. The figure, show that the 8.7 % limit is a low one.

Birmingham samples of skimmed milk were of very variable composition, unwatered samples having solids-not-fat 8.6-9.2 %, and fat 0.4-3.7 % ; some were separated milk and some had not been skimmed. Vendors have described their watered milk as "skimmed" in the vain hope that it would pass as such. No less than half of the samples examined in 1885 were adulterated, some with large proportions of water, and may have been sold to ordinary customers as "milk."

A purchaser of skimmed milk is entitled to at least 1 % of fat. This standard was accepted in a condensed milk appeal case, *Petchley v. Taylor* (1898).

PROSECUTIONS FOR SKIMMED MILK. *Birmingham.* Added water 44 %. Fine £2 (1885 Report).

Glasgow. Fat 0.28 %, being deficient of 72 % of the original fat of skim milk, 1 % being a fair standard. It was admitted to be

machine skimmed. The Sheriff dismissed the case as there was no standard, and as separated milk was fresher (*B.F.J.*, 1900, 335, 369).

Birmingham. Solids-not-fat 7.2 %, fat 2.2 %. Excess water 15 %. It was not skimmed milk, which contains about 1 % of fat. Fine £10 (1901 Report).

London, South-Western. Separated milk. Fine £2 (*B.F.J.*, 1910, 120).

Folkestone. No label on the vessel from which it was sold to show that it was skim milk. Fine £1 (*B.F.J.*, 1926, 88).

Lewes. Deficient in solids-not-fat 11.1 %, which was equivalent to an excess of water of a similar percentage. The milk had been sold in a glass on a racecourse. Fine £20 (*B.F.J.*, 1930, 9).

PROSECUTIONS FOR SEPARATED MILK. *Birmingham.* Deficient of 18 % and 74 % of milk solids, respectively, artificially coloured. The vendor was fined £5 for each sample (1907 Report).

Lambeth. Not separated milk, but a mixture of 58 parts of water with 42 parts of milk, which milk had 10 % of its original fat extracted. Fine £20 (*B.F.J.*, 1907, 89).

Gloucester. Added water at least 10.2 %. Fine £50 (*B.F.J.*, 1929, 89).

Marylebone. Added water 26, 20, and 24 %, respectively. The defence was that the article was pig-wash composed of whey, cream, and sour whole and separated milk. The three churns were labelled "Sep. Milk," which was said to be a railway description. Fine £6 (*B.F.J.*, 1930, 28).

Dudley. Defendant was fined £10 for selling separated milk as "New milk," and 5s. each for three other offences—churn not effectively labelled "Separated Milk," not having his clothing and person clean, and for not keeping clean his vehicle (*B.F.J.*, 1930, 36).

SOUR MILK

Samples of milk as received have usually 15° to 20° of acidity. The rate of increase is very variable; in the same conditions, there may be only a slight increase, or it may be four times as acid the next morning. Experiments by Richmond and Harrison (*S.P.A.*, 1900, 25, 121) indicate the acidity at which milk curdles on boiling is about 33°, that it tastes sour at about 45°, and curdles at about 85° acidity. Richmond and Miller (*S.P.A.*, 1907, 32, 144) have recorded a number of experiments on the relation of preservatives to souring. Richmond and Huish (*S.P.A.*, 1912, 37, 168) have given an improved method of determining the acidity of milk, a table of the average souring of it, and a time-acidity formula.

ANALYSIS. Before analysis sour milks must be reduced to a uniform composition. Lumps may be broken down with a small

whisk made of fine brass wire, or by thorough shaking with lead shot.

The *acidity* is determined on 10 ml. measured in a pipette or cylinder, to which has been added 10 ml. of water, and several drops of phenol phthalein solution. Multiplication by 10 of the number of ml. of N/10 soda or strontia solution used gives the degrees of acidity. The use of a different quantity will alter the figure, which is therefore comparative, not absolute.

For the determination of *total solids* the acidity must be neutralised, phenol phthalein being used as indicator. The weight of the solids must be corrected by the subtraction of the number of ml. of N/10 NaHO used multiplied by 0.0022.

The *fat* may be determined by the Werner-Schmid method, or by putting a quantity of about 11 ml. weighed in a beaker, into a Gerber tube, and washing out the beaker with the sulphuric acid and amyl alcohol; correction must be made for the difference between the weight taken and 11.23 gms.

The elaborate method used in the Government Laboratory has been published by Thorpe (*Trans. Chem. Soc.*, 1905, 206) and it has been criticised by Richmond and Miller (*S.P.A.*, 1906, **31**, 317). In the few Birmingham samples referred, there has been a good agreement between the pairs of analyses.

Nitrogen, the amount of which does not alter on keeping, should be determined in sour milks (Smetham and Ashworth, *S.P.A.*, 1897, **22**, 172).

PRESERVED MILK

As long ago as 1879 (*S.P.A.*, 1879, **4**, 88) Wigner called attention to milk preservatives containing boric acid or borax. In Birmingham, tests were first made for boric acid in 1896, and for formic aldehyde in 1897. In 1900, no less than 11.4 % of the samples of milk contained one of these preservatives. On a few occasions both preservatives were present. The samples of milk from farmers' churns were always free from preservatives, and there was no evidence that they were added at retail shops. The preservatives were therefore added by wholesale dealers or middlemen having a milk round.

In some cases the addition of preservative appears to have been a matter of routine, quite irrespective of the temperature. The average of the months December to February during six years was 3.4 % of preserved samples; August was the highest month with 18 %. During this period seventeen preserved samples were taken on days when the *maximum* temperature did not exceed 39° F. ! (Liverseege, *J. Royal Inst. of Public Health*, 1908, 225.)

The injurious effects on children of milk containing boric acid were demonstrated by a Birmingham case in which a boy of eight

required medical attention after taking such milk ; a girl and a baby in the same family were also ill. In an inquest held at Walthamstow (1904, Aug. 22) the death of a child five months old was considered to be partly due to the presence of formalin in its milk.

Commencing in 1896, Birmingham vendors were prosecuted for the use of boric acid in milk, but in 1897, when the Health Committee cautioned a vendor personally for preserving his milk with formic aldehyde, he informed them he intended to go on using the preservative and that they could not stop him ! Unfortunately that statement was correct, as at that time, while formic aldehyde could readily be detected in milk, there was no available means for determining it and prosecutions could not be instituted unless the amount of adulterant was stated.

As vendors were being prosecuted for the use of boric acid and not for formic aldehyde, the latter preservative became more popular. Between 1897 and 1903 the proportion adulterated with boric acid fell from 5.5 % to 1.5 %, while milks containing formic aldehyde increased from 3.3 % to 6.4 %.

In 1903, the writer having devised a method for the determination of formic aldehyde, there were successful prosecutions for the use of that preservative. During 1903-9, the fines for preservatives in milk amounted to over £42, and in the next five years only 0.2 % of the samples were preserved ; during 1924-8, only one sample of preserved milk was detected, though more than 12,000 were examined.

Most of the above fines were for selling an article not of the nature, substance and quality of the article demanded by the purchaser, but in 1922, when two vendors paid £75 in fines for adulterated milk containing preservative, part of the fines were obtained for offences against the Milk and Cream Regulations, 1912, which specifically prohibit the addition of "any preservative substance to milk intended for sale for human consumption." The Regulations were too late to be of much value in Birmingham, as the action taken during the sixteen years had practically stopped the use of preservatives in milk.

The quantities of preservatives added to milk were often insufficient to make any appreciable difference to its keeping qualities (Richmond, *S.P.A.*, 1900, **25**, 123 ; 1907, **32**, 144). On the other hand, through ignorance or carelessness, large quantities were sometimes present. A Birmingham sample of milk had the equivalent of 130 grains of boric acid per gallon ; it was taken from the bottom of a churn to which *solid* preservatives had been added (*F. & S.*, 1898, Nov. 5). Milk taken from a cart in *January*, 1904, contained 5 parts per 100,000 of formic aldehyde.

Preservatives in milk are also objectionable, as they may be used in substitution for the scrupulous cleanliness necessary in

dairying. Further, while preservatives may retard souring, they may not inhibit the growth of pathogenic micro-organisms.

The use of preservatives for cleansing dairy utensils is prohibited by the Milk and Dairies Order, but is a possible source of their presence in milk.

ANALYSIS. A general method, depending on the decolorisation of litmus, has been given by M. Wynter Blyth (*S.P.A.*, 1901, **26**, 148). Methods for boric acid and benzoic acid have been previously given (pp. 102, 97).

Formic Aldehyde. Although this preservative has been largely used, Richmond considered it of little practical use, as at 80° F. at least 40 parts per million were required to make milk keep twelve hours longer (*S.P.A.*, 1900, **25**, 124). It is destroyed by the tissue oxydases of milk (Schryver, Food Report No. 9 to L.G.B., 1909). After prosecutions for the presence of this preservative, a preparation of it with sodium nitrite was put on the market under the name "mystin," which was incorrectly said to be undetectable (*Analyst*, 1912, **37**, 155, 178).

Its presence is indicated by a violet colour in the Gerber test when the fat is read. The presence of 1 per million may be detected by floating some of the milk on Gerber sulphuric acid containing iron in a test tube. On standing all night a violet ring will appear between the liquids (Hehner's test; cp. Liverseege, *S.P.A.*, 1901, **26**, 152). Larger quantities will show in a shorter time.

For its determination, prepare milk standards by diluting standard formic aldehyde solution with pure milk: 1 ml. diluted to 100 ml. will give 10 per million. Put 10 ml. of the milk and of standards into 1 oz. stoppered bottles, run in (do not drop) 3 ml. of the sulphuric acid reagent, and shake. Compare the violet colours at once and after standing. The standard formalin solution is made by diluting 1 ml. of formalin with 350 ml. of distilled water. It is standardised by adding 20 ml. of about N/10 iodine to 20 ml., then adding 2 ml. 3E.NaOH, followed ten minutes after by 3 ml. 3E.HCl. It is then titrated with N/18.1 sodium thiosulphate solution: 20 ml. of the iodine solution is also titrated, and the difference between the two titrations on multiplication by 0.0415 gives parts of formic aldehyde per 1,000. It is then diluted to contain 1 part per 1,000.

If the milk contains less than 5 parts per million, the method of Shrewsbury and Knapp (*S.P.A.*, 1909, **34**, 12) should be used. Note that their reagent must be used *immediately* after mixing; it becomes useless after an hour or two.

PROSECUTIONS FOR BORIC PRESERVATIVE. *Birmingham.* Boric acid 60 grains per gallon. As it was the first case in Birmingham, the vendor was only fined 1s. and costs (*F.& S.*, 1896, Oct. 24).

Barry. Boric acid 210 grains per gallon. Two vendors were each fined £2 (*F. & S.*, 1897, July 17).

London, Bow Street. Boric acid 33 grains per gallon. The defendants bought their milk from a farmer, who admitted adding to each churn of milk a pint of a preservative solution, which he had made. It contained sugar 6 %, carbonate of soda 3 %, and borax 3 %. The case was dismissed on a warranty (*B.F.J.*, 1899, 86).

Birmingham. Boric acid 7, 7, and 8 grains per gallon, respectively; they were taken simultaneously from the vendor's three shops. The defendant supplied a list of his farmers, but twenty-three samples taken from them were all free from boric acid. Fine £15. A similar case against a customer of his was withdrawn (*B.F.J.*, 1909, 229).

Wigan. Boric acid 5 grains per gallon, and extraneous water. The preservative was stated to be due to the cows' sore udders having been dressed with boric ointment. Vendor ordered to pay costs (*B.F.J.*, 1912, 139).

Chertsey. Boric acid 0.015 %, and 12 % of extraneous water. The defendant suggested that the preservative was due to a preparation used for cleansing his churns. Fine £2 (*B.F.J.*, 1927, 107).

Goole. Boric acid 0.007 %. The farmer admitted having added about a teaspoonful to 10 gallons of milk. Fine £5 (*B.F.J.*, 1928, 107).

PROSECUTIONS FOR FORMIC ALDEHYDE. *Liverpool.* Formalin. One vendor was fined £3 and another £5 (*B.F.J.*, 1900, 262).

Birmingham. Formic aldehyde, 1 and 5 parts per 100,000, respectively. Fine £1 (*B.F.J.*, 1904, 41).

London, Bow Street. Formaldehyde 0.003 %. A Government analyst stated that he had not found it, but that it might disappear in a week. Fine £1 (*B.F.J.*, 1908, 177).

London, South-Western. Formaldehyde and sodium nitrite. It was stated that "mystin" had been used. Fine £5 (*B.F.J.*, 1912, 235).

Birmingham. Formic aldehyde 2 parts per 100,000. Fine £1 (*B.F.J.*, 1927, 116).

Bristol. Formic aldehyde. It was subsequently detected by the Government analysts. The defence suggested that the formic aldehyde had been produced by heat used in the test, but the Public Analyst stated that no heat had been used. Fine £5 (*B.F.J.*, 1930, 17).

PROSECUTION FOR BENZOIC ACID. *London, Marylebone.* Benzoic acid 0.8 grain per pint, contrary to the Milk and Cream Regulations, 1912. Three samples were bought at different times

from the vendor, who was underselling. Fine £10 (*Grocer*, 1913, Nov. 8, 22; *B.F.J.*, 1913, 218).

COLOURED MILK

The addition of colouring matter to milk was first prohibited by the Milk Order, No. 116 of 1920; it was similarly prohibited by sect. 4 of the 1922 Act. The usual additions have been annatto and methyl orange, but Richardson has reported the addition of $\frac{3}{10}$ grain of potassium chromate per pint (Bradford Report, 1903).

The use of colouring matter, though common in some places (Bournemouth had 90 % coloured in 1907), has never been so in Birmingham. It was most frequently detected in 1904-6, when 2.8 % of the samples contained it. A circular sent out by the Health Committee produced a distinct improvement. On one occasion water coloured yellow was found in a milk float, which is no offence. Another vendor was not so wise, he had on his cart on Sunday morning—(1) a churn containing genuine separated milk, (2) coloured separated milk containing 74 % added water, (3) coloured separated milk containing 18 % of water, which was a mixture of the other two ready for sale. The vendor may have been selling the watered separated milk as "milk." He was fined £10. One notable offender in 1904 sold on three occasions, within a month, seven coloured samples of milk containing 10 % to 28 % of water. He sold his milk in a good part of Birmingham and his customers believed it was rich milk. He was fined £100 and left the trade. These cases illustrate the usefulness of the prohibition of colouring.

Methods for the detection of colouring matter in milk have been given by M. W. Blyth (*S.P.A.*, 1902, 27, 146), Cox (*S.P.A.*, 1918, 43, 166), Lowe (*S.P.A.*, 1925, 50, 335), and Gardiner (*S.P.A.*, 1925, 50, 549). Simple methods of testing are as follows:—(1) Put milk into three porcelain dishes, to one add 3E.HCl, to another 3E.NaOH, and compare the colour with the third. Uncoloured milk may give a slight yellow with NaOH. (2) After Leffmann. Put about 20 ml. in a small beaker, add about 1 ml. 3E.Na₂CO₃, put in a piece of white filter about 3 inches by 1 inch, and leave all night. Wash off the milk, put the paper on a white tile, and examine for colour. If annatto be present, stannous chloride produces a permanent red (methyl orange is bleached), and strong H₂SO₄ a blue colour. For prosecutions under the 1922 Act, the certificate need not give a definite quantity of colouring matter.

PROSECUTIONS FOR MILK CONTAINING COLOURING MATTER. *North London.* Skimmed milk to which a colouring matter (annatto) had been added. It was coloured to look like rich Jersey milk. Fines £16 and £35 (*B.F.J.*, 1922, 27).

Birmingham. A vendor, who had been seen to add water and colouring matter to milk, was fined £5 for colouring and £20 for adulterating a sample of milk with 14 % of water (1922 Report).

Bristol. Solid annatto 0.0003 %. Fine £5 (*B.F.J.*, 1924, 37).

Gloucester. Annatto colouring matter equivalent to 0.12 % of Hansen's colouring matter, or 172 parts per million of solid annatto. Fine £1 (*B.F.J.*, 1928, 66).

HEATED MILK. BOTTLED MILK. SCALD MILK

In some prosecutions for sterilised milk it has been suggested that such milk, as the result of the process, is necessarily deficient of fat. During the years 1923-7, 2,530 samples of bottled milk, nearly all sterilised milk, were analysed in Birmingham, and only 0.5 % were deficient in fat : 0.9 % of the milks were deficient in solids-not-fat, most of the deficiencies being small. In several cases the excess of water was traced to a leak in the pasteuriser ; in one instance the leak was so small that it was only the first milk sterilised in the morning that showed adulteration ; water having accumulated in the night.

A Birmingham bottler, two of whose milks had only 1.6 % and 1.9 % of fat, alleged the deficiency was due to fat being deposited on the blade of his new cleaner. This theory was tested at another dairy, and no loss of fat was found by pasteurising, cleaning, and homogenising milk. About 300 gallons of milk yielded under 4 oz. of slime, and it only contained 0.4 % of fat (1930 Report). Any defects in such milk are due, not to the process, but to accident, or adulteration. .

The appeal case *Hennington v. Slater* (*B.F.J.*, 1920, 104) has decided that heating does not remove milk outside the Sale of Milk Regulations ; " hot milk " is still milk.

" Scald milk " is the residual milk after making clotted cream.

Weinstein (*Analyst*, 1929, **54**, 237) and Rothenfusser (*Analyst*, 1930, **55**, 758 ; 1931, **56**, 747) have proposed several methods for giving an indication of the extent to which milk has been heated.

Unheated milk should contain at least 0.4 % of lactalbumin, and sterilised milk will be practically free from it. It may be determined by Leffman and Beam's modification of Sebelien's method. To 25 ml. of milk (neutralised if distinctly acid), add 50 ml. of a saturated (about 1 in 1) solution of pure magnesium sulphate and 24 gm. of the solid salt. Shake in a stoppered cylinder, allow to stand all night ; measure the volume, shake and pour into a large filter. Measure the filtrate, add 3 gm. phosphotungstic acid, and allow to stand all night. Filter, wash once with saturated magnesium

sulphate solution, and determine the nitrogen in the residue on the filter.

$$w/v \text{ lactalbumin} = \frac{0.0356 \, n [T - (0.275 \, F + 0.51)]}{P},$$

where, n = ml. N/10 HCl neutralised, F = % fat in milk, T = total volume and P = volume of filtrate.

PROSECUTIONS FOR STERILISED MILK. *North London.* Fatty solids 18 % deficient. The prosecution was dismissed as the deficiency was considered to be due to the heating (*B.F.J.*, 1905, 228).

West Ham. Non-fatty solids 9.9 %. Fine £5 (*B.F.J.*, 1912, 76).

PROSECUTION FOR HOT MILK. *London, Old Street.* Extraneous water 13 %. It was being supplied to schoolchildren. Fine £2 (*B.F.J.*, 1912, 34).

PROSECUTION FOR HEATED MILK SOLD AS FRESH MILK. *Norwich.* Not of the nature, substance and quality demanded, it having been heated to at least 180° F. It was stated that pasteurisation as recognised by the Ministry of Health was heating to 145–150° F., and that process diminished or destroyed the vitamin C content of the milk. The milk in question had been heated to a higher temperature and should not be sold as “fresh milk.” Evidence was given in support of this. For the defence, evidence was given by the President of an important Dairymen’s Society that for twenty-five years it had been the universal practice of the trade to heat milk to a varying degree, sometimes to 180°, and sell it as fresh, so long as it was sweet and palatable. The chairman said: “In the light of the evidence we cannot say it was not fresh milk, and the case is dismissed” (*B.F.J.*, 1931, 109).

PROSECUTION FOR BOTTLED MILK. *Birmingham.* Solids-not-fat deficient 13 %. Fine £1 (1924 Report).

PROSECUTIONS FOR SCALD MILK. *Plymouth.* Extraneous water 23 %, 24 %, and 14 %, respectively. The Public Analyst gave evidence that he had calculated the water on 8.5 % of solids-not-fat, though he might have used a higher figure, as 10 % of water was lost by evaporation during the process. Dairy managers gave evidence that they added no water when scalding milk. As they were the first prosecutions a nominal fine of 1s. each, and costs, was inflicted (*B.F.J.*, 1908, 214).

Devonport. Extraneous water 9 %. Fine £10 (*B.F.J.*, 1911, 159).

DIRT IN MILK

Dirt in milk is obviously undesirable, not merely as dirt, but as an indicator of bacterial pollution. That its presence in town milk in more than traces is unnecessary, is shown by figures given by Elsdon for Salford in his Report for 1915. During seven years

2,765 samples were examined for dirt ; 34.2 % of them contained none, 43.7 % 1 part per 100,000, and 7.6 % 3 parts. One dirty milk contained 90 parts ! See also Tankard (*B.F.J.*, 1919, 61).

ANALYSIS. Methods have been given by Lowe (*Chemical News*, 1912, 61 ; *Analyst*, 1912, **37**, 450) and Tankard (*S.P.A.*, 1923, **48**, 444 ; 1926, **51**, 31).

PROSECUTIONS FOR DIRT IN MILK. *Hull.* Cow dung 33.6 grains per gallon. Fine £20 (*B.F.J.*, 1911, 36).

East Riding. Dirt 14 parts per 100,000. The Government analysts subsequently found 1.8 parts only, and the case was dismissed, the defendant being allowed 5 guineas costs (*B.F.J.*, 1911, 137).

Hull. Dirt 9 parts per 100,000, exceeding the limit of 4 parts ; of the sediment, 2 parts were cow dung which should not be present. Fine 2 guineas (*B.F.J.*, 1913, 57).

Appeal case *Kenny v. Cox* (1921), " New milk of good quality " was asked for. It was found to contain " a considerable quantity of dirty *débris*," and therefore was not a " milk of good quality." The case was dismissed by the magistrates and the High Court confirmed. It was held to be an attempt to set up an impossible standard ; the impurities being present in so small a quantity that a public analyst could not use terms which would convey to an ordinary lay individual the result of his analysis.

Ulverston. Cow dung 70 parts by volume per 100,000 volumes of milk. Fine £2 (*B.F.J.*, 1922, 118).

Hull. Extraneous solid matter (dirt) as a sediment, consisting partly of dung, to the extent of 3.7 parts by volume of moist sediment per 100,000 parts of milk. The Government analysts, who examined the sample when it was six weeks old, found there had been considerable decomposition. The grit, starch and vegetable fibre amounted to 3 parts per 100,000, and there were also 16 parts per 100,000 of an insoluble proteid substance. Fine 5 guineas (*B.F.J.*, 1923, 17).

Kingston. Exposing for sale milk which was unwholesome, and unfit for food, contrary to the Public Health Act. Evidence was given that in each of thirty-two bottles of milk dirt was visible. Fine £10 (*B.F.J.*, 1930, 7).

CONDENSED MILK

In 1911 the L.G.B. published a lengthy report (Food Report No. 15) by Coutts on condensed milk, and its use as an infants' food, and in 1914 another (No. 21) by Delèpine on its bacterial contents. Later work by Savage and Hunwicke is given in Special Report No. 13 (1923) of the Food Investigation Board of the Department of Scientific and Industrial Research.

The first samples bought in Birmingham were in 1890, and one of them, described as "partly skimmed," was labelled, "Add 4 or 5 parts of water to 1 of milk; If less water is added it may be used instead of cream." The condensed milk contained only 2 % of fat, and to suggest that such a liquid after dilution could be used instead of cream was grossly misleading, as undiluted it contained about half the fat of ordinary *milk*.

Some samples, containing 0.4-0.5 % of fat, examined in 1916, were directed to be diluted with 5 parts of water to 1 of milk.

Some of the full-cream condensed milks were of good quality, containing 11 % or more of fat, but others had been partly skimmed, though the fact was not declared on the label. One sample containing only 8.4 % of fat was labelled, "Only a small quantity of pure sugar added." The small quantity amounted to 40 % !

The 1899 Act, sect. 11, required skimmed milk to be labelled as such, and in 1923 the Condensed Milk Regulations provided limits of composition and standard labels. Fresh Regulations, which came into force in 1927, required that the words "Unfit for Babies" applied to machine-skimmed condensed milk should be brought into greater prominence (Regulations given, *Analyst*, 1923, **48**, 272; 1928, **53**, 98).

To ascertain if a tin of condensed milk complies with the Regulations the following observations must be made :—

1. Weight in oz. Total.
2. ,, ,, Tin.
3. ,, ,, Nett.
4. Label. Stated weight, oz.
5. ,, Declaration, conformable to Regulations ?
6. ,, ,, pints of milk.
7. ,, Name and address of manufacturer or dealer.
8. ,, *Improper reference to quality, etc. (sect. 6).*
9. ,, “ Milk,” unqualified by “ Skimmed ” or
 “ Machine-skimmed ” (sect. 7).
10. Exposed for sale in unmarked wrapper ?
11. Analysis. Total solids %.
12. ,, Sucrose %.
13. ,, Milk solids %. (31 % required for full cream,
 and 26 % for sweetened
 machine-skimmed).
14. ,, ,, oz. in tin, calculated.
15. ,, ,, ,, found.
16. ,, Fat % (9·0 % required for full cream).
17. ,, ,, oz. in tin, calculated.
18. ,, ,, ,, found.

19. Analysis. *Milk solids-not-fat* %.
20. „ „ „ oz. in tin, *calculated*.
21. „ „ „ „ *found*.
22. „ Sp. gr. (if dilution directions).
23. Dilution directions. Parts of water to 1 part of milk.
24. „ „ Milk by volume ?
25. „ „ Of equivalent composition ?
26. „ „ *Milk solids in mixture* % (12·4 % required).
27. „ „ *Fat in mixture* % (3·6 % required).
28. „ „ *Milk solids-not-fat in mixture* % (9·0 % required).

Determinations of ash, hydrated lactose, and boric acid may be required.

Observations Nos. 8, 9, 10, 19, 20, 21, and 28 are not required for full-cream milks, and Nos. 14, 15, 17, 18, 26, and 27 are not required for skimmed milks. For unsweetened milks, Nos. 11 and 12 are unnecessary.

ADULTERATION. There have been few offences against the Regulations in the forty full cream and the sixty-five machine-skimmed condensed milks examined in Birmingham. In three samples the amounts of fat were slightly low (8·7–8·9 %) ; in two cases there was a failure to state that diluted milk prepared according to the directions was not of equivalent composition to milk. In one case milk prepared according to directions was too low in milk solids (12·1 %) and fat (3·2 %). The makers argued that the calculation should be made on the diluted milk, *apart* from the added sugar, and not on the total diluted milk *including* the sugar, and that their directions were therefore correct. I could not agree with this argument.

One sample of machine-skimmed condensed milk was improperly stated to be “ of the highest quality ” ; another sample was described as “ milk ” ; and another as “ skimmed ” instead of “ machine-skimmed ”—it had only 0·4 % of fat : a condensed “ skimmed ” milk should have at least 3·5 % of fat. Apparently no “ skimmed ” condensed milk is on the market, and so the failure of the Regulations to make the distinction is immaterial.

During 1901–3, 2·6 % of the samples examined in England and Wales were reported adulterated, and 2·3 % of those during 1919–30.

ANALYSIS. The Standing Committee convened by the Milk Products Committee of the Society of Public Analysts and Other Analytical Chemists has published reports on the analysis of condensed milk. The first report (*S.P.A.*, 1927, **52**, 403) dealt with the determination of total solids, and of fat, and the second (*S.P.A.*, 1930, **55**, 111) gave a lengthy investigation on the determination of

sucrose. A Report (No. 57) on the Determination of Sucrose, Lactose and Invert Sugar in Sweetened Condensed Milk has been published by the Ministry of Health (review, *Analyst*, 1930, **55**, 573). The calculation of the concentration of condensed milk has been discussed by Allen (*S.P.A.*, 1896, **21**, 281) and McGill (*S.P.A.*, 1898, **23**, 128). See also under "Sugar" (p. 155, f.).

CALCULATION. When the percentage composition and the weight of the contents of a tin have been determined, the weight in ounces of the constituents can be determined and compared with the weight required by the particular number of pints stated on the label, given in the table below. Milk is presumed to have a sp. gr. of 1.032, and machine-skimmed milk 1.035, on a basis of 8.9 % solids-not-fat, and 0.1 % fat.

Pints	$\frac{3}{4}$	$\frac{7}{8}$	1	$1\frac{1}{2}$	$1\frac{5}{8}$	$1\frac{3}{4}$	$1\frac{7}{8}$	2
MILK (FULL CREAM).								
Milk solids, oz.	1.91	2.23	2.55	3.83	4.15	4.47	4.79	5.11
Milk fat, oz.	0.55	0.64	0.74	1.11	1.20	1.30	1.39	1.48
MACHINE-SKIMMED MILK.								
Milk solids-not-fat, oz.	1.39	1.63	1.86	2.79	3.02	3.26	3.49	3.73

For example, a condensed milk contained 34.8 % of milk solids and 9.8 % of milk fat and was labelled to be equivalent to $1\frac{3}{4}$ pints of milk; the weight of the contents was 13.8 oz. It therefore contained 4.80 oz. of milk solids and 1.35 oz. of milk fat, and was in excess of the requirements of 4.47 oz. and 1.30 oz., respectively, shown in the above table.

The percentage of a constituent in a milk diluted according to the directions on the label is $\frac{S \times P}{S + V}$, where S is sp. gr. of the condensed milk, P the percentage of the given constituent, and V the volume of water directed to be added to 1 volume of milk (Liverseege, *S.P.A.*, 1924, **49**, 276). Other methods of calculation have been given by Hinks (*S.P.A.*, 1923, **48**, 596) and Essery, with tables (*S.P.A.*, 1924, **49**, 178).

PROSECUTIONS FOR CONDENSED MILK. *Glasgow.* Deficient of 44 % of natural fat. The tin was labelled, "This milk is guaranteed to contain all its original cream." The Sheriff dismissed the case, first, because no standard was given on the certificate, and also, because "Cornet Brand" was asked for, and as the inspector received "Cornet Brand" he was not prejudiced (*F. & S.*, 1895, Oct. 19, 26).

Blaenavon. Entirely separated milk. It was labelled "Full cream" and "all the original cream." Fine £10 (*F. & S.*, 1898, June 18).

Petchley v. Taylor (1898). This appeal case arose from a magistrate convicting a vendor for selling a condensed milk deficient of 97 % of its original fat. The tin was labelled "Skimmed milk

with nothing added but the finest sugar." Evidence had been given that by skimming the surface of the milk not more than 63 % of the fat could be removed. The article had been prepared from separated milk. The High Court dismissed the appeal (*Analyst*, 1898, **23**, 168).

Glenarm. Only 1.4 % of fat instead of at least 9.96 %. At the time of purchase the inspector did not know that the tin was marked "Condensed machine-skimmed milk." The defendants were convicted, and appealed to the King's Bench Division, Dublin. The Court upheld the decision of the magistrates, stating that the article supplied was not of the quality generally understood by "condensed milk" (*Grocer*, 1906, Jan. 13, May 26).

North London. Only 0.61 % of cream, and also, not labelled "Machine-skimmed milk." Fine £1 (*Grocer*, 1914, March 14).

Yeovil. Fatty solids 5 % deficient, but a second purchase gave different results. The case was withdrawn, as there was a doubt if the thorough mixing of the contents of the tin had been done (*B.F.J.*, 1925, 15).

Exeter. "Full cream" containing only 26.5 % milk-solids, while fat was only 8.5 %. Fine £2 (*B.F.J.*, 1925, 35).

Rotherham. The contents of the tin were equivalent to 1.68 pints of ordinary milk, instead of 2 pints as stated. The tin only contained 14 oz., probably due to a defect in the plunger which measured the milk. Fine £20 (*Analyst*, 1926, **51**, 459; *Grocer*, 1926, July 17).

Stockport. Exposing for sale condensed milk in unlabelled tins. Fine £5 (*Grocer*, 1930, June 7; *B.F.J.*, 1930, 68).

DRIED MILK

In 1918 the L.G.B. published a lengthy report (Food Report No. 24) on an Inquiry as to Dried Milk, with Special Reference to its use in Infant Feeding, by Coutts, Winfield, and Monier-Williams. See also Richmond (*S.P.A.*, 1906, **31**, 219).

In 1923 the Ministry of Health published Dried Milk Regulations which prescribed limits for five qualities of dried milk, and required suitable directions to be on each packet (*Analyst*, 1925, **50**, 343). Further Regulations in 1927 (*Analyst*, 1928, **53**, 98) required the words "Unfit for Babies" to be made more prominent.

Of the samples of dried milk examined in England and Wales, 1927-30, 2.7 % were adulterated, and of the few examined in Birmingham, one "full cream" had only 24.6 % of fat and was not properly labelled, and one "skimmed milk" containing only 0.7 % of fat was labelled "It won't feed babies. For every other milk purpose it is more convenient than ordinary milk"!

ANALYSIS. On pp. 177-184 of the Report mentioned above the methods used in the Government Laboratory are given, and

analyses of samples on pp. 36 and 157. Later, Jephcott investigated the estimation of fat, lactose and moisture (*S.P.A.*, 1923, **48**, 529). Salamon (*S.P.A.*, 1924, **49**, 170) stated that the Rose-Gottlieb method for fat invariably gave lower figures than the Werner-Schmid one. Lampitt and Hughes have given a method for estimating the insoluble portion of dried milk (*S.P.A.*, 1924, **49**, 176), and Lampitt and Bushill have studied the effect of the water content on solubility (*S.P.A.*, 1931, **56**, 778).

CALCULATION. Methods for the calculation of "equivalent pints" have been given by Hinks, and also by Henville (*S.P.A.*, 1924, **49**, 471 and 472).

PROSECUTIONS FOR DRIED MILK. *Westminster.* Fat 16·3 % instead of 25 %. Fine 10s. and 2 guineas costs (*Grocer*, 1905, May 20).

East Ham. Separated milk. It was labelled "Pure milk powder." Fine £10 for each of two samples (*Grocer*, 1918, March 9 ; *B.F.J.*, 1918, 46).

Sheffield. Selling dried skimmed milk intended for human consumption which was not contained in a properly labelled receptacle. Fine £1 for each of two cases (*B.F.J.*, 1927, 105).

BUTTERMILK

Hodgson (*S.P.A.*, 1919, **44**, 229) has published tabulated analyses of 312 samples of buttermilk bought in Manchester. Prosecutions have taken place when the added water exceeds 30 % (solids-not-fat 5·95 %), and cautions when the addition has been between 25–30 %. He concluded that it is possible to produce buttermilk in practically every month of the year, without the addition of any water whatsoever. The fat, under good conditions, should not exceed 0·6 %.

PROSECUTIONS. *BELFAST.* Solids 6·5 %, indicating 23 % excess of water. Fine £5 (*Analyst*, 1877, **1**, 81).

Excess added water 44 %. The solids were only 4·5 %, instead of 8 %, which allowed 20 % of added water for churning. Fine £3 (*B.F.J.*, 1901, 131).

Added water 42·5 %, being 22·5 % in excess of the 20 % allowed for churning. Fine £2, confirmed on appeal to the Recorder's Court (*B.F.J.*, 1901, 209).

Selling a mixture of skimmed milk, water and lactic acid, to which a false trade description "buttermilk" had been applied. Fine £5 (*B.F.J.*, 1929, 46).

CHAPTER XVII

ANALYSIS OF OILS, FATS AND WAXES

Titre, melting-point. Refractive index. Valenta number
Saponification value. Acid value. Unsaponifiable matter. Reichert,
Polenske, Kirschner values. Soluble and insoluble fatty acids. Iodine
and bromine absorptions.

IN addition to books dealing specially with this subject, the following should be consulted. The Report by the Committee of Analysts to the Ministry of Food on Standard Methods (*Analyst*, 1920, **45**, 278). The Committee also suggested standards for oils of "good merchantable quality" (p. 288). Bolton and Revis criticised various methods for the analysis of arachis and sesame oils, butter, etc. (*S.P.A.*, 1915, **40**, 499). Christian and Hilditch have published a research on the estimation of saturated glycerides in fats (*S.P.A.*, 1930, **55**, 75). The methods given in the B.P. should also be consulted.

TITRE AND MELTING-POINT

The following methods are convenient when great accuracy is not required.

Titre. Half fill a beaker (2 inches by 1 inch) with the melted fat and stir with a thermometer graduated to 0.5°C . The readings are recorded each half-minute, until they are constant, or there is a rise of temperature. The highest point of the rise, or the stationary point, is the titre. The method is often used for fatty acids.

Melting-point. Using the same beaker and thermometer, warm slowly while stirring on a copper tray over boiling water till clear. With lard the determination may be repeated without an interval.

REFRACTIVE INDEX

The Zeiss butyro-refractometer is a convenient instrument for an oil or fat the refractive index of which lies between 1.422 and 1.489. Enough oil or melted fat to cover the prism (about 2 drops) must be used, and after waiting a minute or two the reading of the scale and of the thermometer is taken. For butter, margarine, etc., white light may be used, but for other liquids the use of a sodium flame will prevent a coloured fringe. The setting of the scale should be periodically tested with liquid paraffin, refractive index 1.474, which

should give scale reading 57.7 at 40° C. or 69.5 at 20° (Liverseege and Singleton, *S.P.A.*, 1921, **46**, 93).

The relation between the scale reading and the refractive index has been given in complicated formulæ by Roberts (*S.P.A.*, 1916, **41**, 376), Liverseege (*S.P.A.*, 1919, **44**, 49), and Richmond (*S.P.A.*, 1919, **44**, 167), but it is simpler and more accurate to use the table furnished by the makers of the instrument, or, better still, the extension of it given by Evers and Elsdon ("Analysis of Drugs and Chemicals," 340).

For the examination of waxes, Marpmann (*Analyst*, 1901, **26**, 217) has suggested the admixture with a fatty oil, and has stated that the R.I. of a mixture is equal to the arithmetic mean of the R.I. of its components. The following test was made of this statement:—One volume of mineral oil was mixed with 1.02 volumes of almond oil (R.I. 1.4699 at 25° C), and the R.I. of the mixture was 1.4779 at 25° C.

R.I. of mixture $\times 2.02 = 2.9853$, from which is subtracted R.I. of almond oil $\times 1.02 = 1.4993$, and the difference is 1.4860, while the actual refraction of the mineral oil was 1.4858. This method is useful for liquids which have R.I. somewhat outside the range of the scale.

The temperature of 25° C. has been used for the refraction of oils, but some fats are not melted at 25°, and, as calculations are sometimes made of mixtures of oils and fats, the uniform temperature of 40° is better. Further, in summer, it is easier to maintain a constant temperature of 40° than of 25°. Volatile oils are better examined at 20° or 25°, and waxes and hard fats at 60°.

A number of authors have investigated the effect of temperature. Tolman and Munson (*Analyst*, 1902, **27**, 298), Leach and Lythgoe (*Analyst*, 1905, **30**, 176), Richmond (*S.P.A.*, 1907, **32**, 44), Wright (*Analyst*, 1920, **45**, 52), Joseph (*Analyst*, 1920, **45**, 182). From these papers and the author's results, it appears that there is a constant variation in the refractive index of 0.00036 for 1° C. This figure is useful for making small corrections when the temperature of observation is near, but not exactly, 40°; and, although the variation in scale readings is not similarly constant, the use of the mean value of 0.6 for 1° C. will not introduce any serious error in these conditions. For larger corrections, such as calculating the reading at 40° from that at 25°, a graphical method has been given (p. 111), or the following table may be used, or 0.0054 subtracted from the refractive index:—

TEMPERATURE CORRECTION OF REFRACTOMETER SCALE

Scale reading at 25° C.	40	45	50	55	60	65	70	75	80	85	90	95
Subtract to get reading at 40° C. .	7.7	7.9	8.1	8.3	8.5	8.7	8.9	9.2	9.4	9.6	9.8	10.0

Some essential oils, as turpentine and sandalwood, have low temperature coefficients (G. Thompson, *S.P.A.*, 1922, **47**, 469; Harvey, *S.P.A.*, 1923, **48**, 19). Freyer and Weston have given figures obtained with a number of fixed oils and commercial hydro-carbons, and also dispersive powers. They found that free acid lowered the refractive power, and that there was an increase on heating some oils (*S.P.A.*, 1918, **43**, 312). Evers and Elsdon have determined the refractive indices of ointments and ointment material at 60° C. (*S.P.A.*, 1922, **47**, 200). Long has given the refractions of several vegetable oils at different temperatures (*Analyst*, 1890, **15**, 34).

VALENTA NUMBER

Take equal volumes of the clear filtered fat and Valenta acetic acid (about 3 ml.), heat in a test tube, stirring with a thermometer graduated to 120° C., till the mixture is clear. The liquid is allowed to cool, while stirring with the thermometer, until it is opalescent, when the temperature is read. A second reading may be taken. If the fat used is rancid, or it has been heated long, a low reading may be obtained. The presence of water must be carefully avoided.

The acid used should be of such a strength that it indicates about 110° C. with a mixture of lards, and about 60° C. with a mixture of butters. It corresponds to B.P. glacial acetic acid with about 0.2 v/v of water added, but it is more conveniently made by adding 1 ml. 10E.H \bar{A} to 100 ml. of glacial acid, and adding one, or other, of the acids as is shown to be necessary by trial.

Fryer and Weston have published a detailed investigation of the test (*S.P.A.*, 1918, **43**, 3). They prefer almond oil for standardising the acid. To avoid the freezing of the acetic acid, Parkes (*S.P.A.*, 1918, **43**, 82) has suggested the addition of propionic or butyric acid to the acetic acid.

SAPONIFICATION VALUE

This was defined by Koettstorfer, the originator of the process, as "the number of mgm. of KOH which will saponify 1 gm. of fat" (*Analyst*, 1879, **4**, 106). This statement is unnecessarily complicated as analyses of oils are rarely made for their use as soft soap, when that statement might possibly be useful. The writer prefers the simpler statement giving the number of ml. of N alkali for the saponification of 100 gm. of oil or fat, which can be expressed as N v/w. Division of the saponification value by 0.56 will give the simpler statement.

Increased accuracy may be given to the method by taking 5 gm. of oil and 50 ml. of N/2 alcoholic KOH, instead of the 1.5 or 2 gm. and the 25 ml. of the B.P. 1914. The oil is weighed in a wide-mouth resistance glass flask, 50 ml. of the alkali added from a delicate

pipette, draining for a definite time; the flask is covered with a watch glass, and heated by putting it on the hot metal over a water-bath or water-oven, with occasional agitation for a little time after solution is complete. With waxes which require vigorous boiling a metal ball-condenser is advisable. The solution is titrated with N/2 HCl after addition of phenol phthalein. As the strength of the alkali rapidly falls in the presence of the fat, the writer prefers to use for calculation the mean between a hot and a cold blank.

$$\text{Saponification value} = \frac{28 (\text{ml. used in blank} - \text{ml. used for oil})}{\text{weight of oil}}$$

$$N \text{ v/w} = \frac{50 (\text{ml. used in blank} - \text{ml. used for oil})}{\text{weight of oil}}$$

The alkaline solution is conveniently made by diluting 10E.KOH with spirit, and filtering the next day. The spirit used is prepared by leaving two sticks of caustic potash in a W.Q. of industrial spirit for a few weeks, then redistilling.

For substances difficult of saponification, such as wool fat and beeswax, Pardee, Hasche and Reid consider butyl alcohol as solvent to be better than ethyl alcohol (*Analyst*, 1920, **45**, 268). Fryer found that the use of amyl alcohol doubled the velocity of the saponification (*S.P.A.*, 1921, **46**, 89).

Koettstorfer introduced his method for the analysis of butter, which has little acidity, but for balsams and beeswax the "ester" value should also be determined; it is the difference between the saponification and acid value.

ACID VALUE

With oils, where the acidity is small, 10 gm. may be taken, warmed with 50 ml. of neutralised purified methylated spirit, phenol phthalein added and titrated with N/10 NaOH. With beeswax 5 gm. should be taken and titrated with *alcoholic* N/2 KOH, until the red is permanent on short heating: 50 ml. of the alkaline solution should then be added and the ester value be determined. An unsaponifiable residue will indicate paraffin.

UNSAPONIFIABLE MATTER

Weigh about 10 gm. in wide-mouth flask, add 50 ml. spirit, 5 ml. 10E.NaOH, and heat on water-bath until residue ceases to decrease. Evaporate off alcohol in basin on water-bath, and dissolve soap in 200 ml. water. Shake out with 50 ml. of ether, followed by 25 ml. twice. If the separation is poor, add alkali, or alcohol and ether. The mixed ethereal liquids, after washing three times with a little water to remove soap, are transferred to a weighed flask, the

ether distilled off, and the residue dried and weighed. The B.-R. value of the residue should be taken if possible.

Wilkie (*S.P.A.*, 1917, **42**, 200) has made suggestions for the prevention of emulsification, and the addition of castor oil for beeswax. Steuart has studied the sterols of edible fats (*S.P.A.*, 1923, **48**, 155), and Lester Smith, the determination of unsaponifiable matter (*S.P.A.*, 1928, **53**, 632; 1931, **56**, 9). Bolton and Williams found that the amount of iodine absorbed by the unsaponifiable matter divided oils into four groups. They have also given a method for its determination (*S.P.A.*, 1930, **55**, 5). A. More has given a Dutch method for separating sterols from small quantities of butter (*S.P.A.*, 1929, **54**, 735).

Shrewsbury detects paraffin wax in lard by saponifying 5 ml. with 20 ml. glycerol soda and adding to the hot mass, drop by drop, 50 ml. of industrial methylated spirit, while shaking. If 2 % of paraffin wax be present, the solution will be cloudy on cooling (*S.P.A.*, 1909, **34**, 348; 1914, **39**, 296). Dunlop also has written on the detection of paraffin products in lard and margarine (*S.P.A.*, 1909, **34**, 524).

REICHERT, POLENSKE AND KIRSCHNER VALUES

In the original Reichert method, 2.5 gm. of the fat was saponified with alcoholic KOH solution (*Analyst*, 1885, **10**, 104); Meissl increased the quantity of fat to 5 gm. Wollny in a classic research (*Analyst*, 1887, **12**, 203, 235; 1888, **13**, 11, 38) advocated the substitution of NaOH, and precautions to avoid the absorption of CO₂. His method and apparatus were modified and standardised by a committee of analysts (*S.P.A.*, 1900, **25**, 309). To facilitate the process and to avoid possible formation of acetic acid Leffmann and Beam recommended the use of glycerin instead of alcohol (*Analyst*, 1891, **16**, 153; 1892, **17**, 65); the latter, as described below, is the one by which the writer's figures have been determined, the standard apparatus (*op. cit.*) being used. The Reichert and Polenske figures of a number of oils have been determined by Elsdon and Hawley (*B.P. Conf.*, 1924, 573).

Reichert. Weigh 4.95–5.05 gm. of the clear fat, or measure with a 100 grain pipette calibrated to deliver about 5 gm. of melted fat (about 5.8 ml.), into a 300 ml. resistance glass flask. Add 15 ml. of 2.2E. glycerol soda, and, if the Polenske is to be determined, 4 ml. of glycerol. Heat over naked flame, shaking till frothing has ceased and the liquid is clear. Add 135 ml. (or 131 ml. if the Polenske is being determined) of boiled hot water gradually, while shaking, from a separator, then 10 ml. of 3.6E. H₂SO₄. To prevent bumping add about 0.1 gm. of pumice in No. 40 powder, and a few drops of indicator to show that the liquid is acid. Distil 110 ml. in nineteen

to twenty-one minutes. The asbestos shield should fit the flask (Cocks and Nightengale, *S.P.A.*, 1928, **53**, 322).

Put the flask in water about 15° C. for at least ten minutes (it must not stand overnight). Shake and filter through 11 cm. dry neutral filter into 100 ml. flask, reject the first 5 ml., and titrate 100 ml. with N/10 NaOH, using phenol phthalein as indicator. No water is to be used but the partly titrated liquid used for washing out the flask. After subtracting the blank, the figure is increased by one-tenth and corrected to 5 gm. if necessary. The titrated liquid may be used for Kirschner. The blank is the titration of 100 ml. of distillate treated as above, but without any fat. A figure much over 1 ml. may be due to impurity in the glycerin.

Polenske (*Analyst*, 1904, **29**, 154; 1911, **36**, 335; 1920, **45**, 223; *S.P.A.*, 1920, **45**, 293). Put the 110 ml. flask under the condenser tube, and wash the tube with 18 ml. distilled water. Pour these washings on to the filter paper and reject the filtrate. Wash the condenser tube, flask and filter paper successively with 20, 15 and 10 ml. of methylated spirit, neutral to phenol phthalein, putting a cylinder under the condenser tube when the flask is not there. Add phenol phthalein solution to the filtrate and titrate with N/10 NaOH. Correct to 5 gm. if necessary.

Kirschner (*Analyst*, 1905, **30**, 205; 1911, **36**, 336; *S.P.A.*, 1928, **53**, 322). Dilute the neutralised Reichert filtrate to 110 ml. (or other convenient volume) and add 0.5 finely powdered Ag_2SO_4 , when coconut oil will yield a precipitate of silver caprylate. Allow to stand for an hour with occasional shaking. Filter, take 100 ml., add 42.5 ml. of boiled water and 2.5 ml. of 3.7E. H_2SO_4 . After adding about 0.1 gm. of pumice in No. 40 powder, distil 110 ml. Filter, titrate 100 ml. with N/10 NaOH, and subtract the blank. Correction for dilution: (a) If diluted to 110 ml. before distillation Kirschner = ml. \times 4/3. (b) If the volume was "*n*" ml. Kirschner = ml. \times *n* \times 0.0121. Correct the result to 5 gm. if necessary.

SOLUBLE AND INSOLUBLE FATTY ACIDS

These were important early methods for detecting foreign fat in butter (Muter, *S.P.A.*, 1877, **1**, 8; Dupré, *S.P.A.*, 1877, **1**, 87; Jones, *S.P.A.*, 1878, **2**, 19, 37).

Prepare E/2 alcoholic NaOH by adding 10E. NaOH to industrial methylated spirit in the proportion of 5 to 95. Allow to stand overnight and filter. Weigh, or measure, 5 gm. of the fat into a 300 ml. resistance glass flask and pipette 50 ml. of the alcoholic soda to each fat, draining a definite time, and to two other flasks for hot and cold blanks, cover with watch glasses. Leave on copper tray over boiling water, with an occasional shake, till some time after solution is complete, say one and a half hours.

Add to each flask about 150 ml. of boiling water. Pipette 50 ml. of N/2 HCl and fill the flasks up to the neck with boiling water. The saponification value may also be obtained if the liquid be titrated with N/2 HCl, but the amount used for the fats must be made up to that used for the blanks. The flasks are left on the tray till the acids have mostly separated and make a clear layer above the aqueous liquid. Allow them to cool and stand overnight.

Filter through a 12.5 cm. filter paper, and wash the fatty acids on the filter paper once with hot distilled water. Add phenol phthalein to the filtrate and titrate with N/10 NaOH. Wash the fatty acids in the flasks with about 100 ml. boiling water, cool, filter and titrate. Subtract the mean number of ml. used by the blanks from the amount used by the butters, and, if necessary, correct to 5 gm. The soluble acids are usually expressed as butyric acid (1 ml. N/10 = 0.0088).

The percentage of insoluble acids (Hehner number) is obtained by draining the cake in the flask, and dissolving it and that on the filter in alcohol and ether. Evaporate in a weighed flat-bottom glass basin, adding absolute alcohol if water separates. Dry on the tray till constant, weighing each half hour.

IODINE AND BROMINE ABSORPTION. The method of Hübl, using an alcoholic solution of iodine and mercuric chloride (*Analyst*, 1885, **10**, 9), has been shown to depend on the action of iodine chloride (Wijs, *Analyst*, 1898, **23**, 240), and has been practically superseded by that of Wijs.

The Wijs solution is a N/5 solution of iodine chloride in glacial acetic acid, though 30 v/v of carbon tetrachloride may be present to prevent crystallisation. The B.P. 1914 prepared it from iodine and chlorine following earlier directions (Wijs, *Analyst*, 1898, **23**, 240), but the author's later directions are to use a 9 w/v solution of iodine trichloride and add iodine (Wijs, *S.P.A.*, 1929, **54**, 12; also 1928, **53**, 656). It is convenient to use the contents of an 8 gm. sealed tube of iodine trichloride for about 880 ml. of glacial (at least 99 %) acetic acid. When solution is complete 5 ml. are mixed with 10 ml. of E.KI and 50 ml. of water and titrated with thiosulphate solution; about 8.8 gm. of powdered iodine are then added to the remainder. When most of it is dissolved, the clear liquid is again titrated and the solution of the iodine stopped by filtration if the second titration is one and a half that of the first. There must not be an excess of iodine trichloride. The solution may be diluted to about N/2, but it is desirable each time to titrate the exact quantity used. As the end point is delicate and as the factor for N/10 iodine is an awkward one, the writer prefers to use a N/18.13 solution of thiosulphate, of which 1 ml. = 0.007 iodine.

Wijs states that a large excess is necessary; not more than 30 % of the total amount present should be absorbed. The weight of oil

taken for a determination should not be greater than the weight found by dividing the highest probable iodine value into 19 gm. For instance, cod-liver oil (190) would require 0.1 gm.

The oil is weighed in a small crucible or watch glass, which is put in a wide-mouth stoppered flask, and dissolved in 10 ml. of carbon tetrachloride. With waxes more of the solvent may be required and gentle heating : 25 ml. of the solution is added from a delicate pipette, allowing to drain a definite time, and using a piece of rubber tubing to prevent any of the corrosive liquid going into the mouth. The flask is left in the dark for one hour for most oils, but two hours for linseed and fish oils. At the end of the time, 10 ml. of E.KI is run over the stopper to remove any iodine ; then are added 100 ml. of water and 50 ml. of the thiosulphate solution from a pipette, and the titration completed. The blank containing 25 ml. of the solution with 10 ml. of carbon tetrachloride, is treated similarly, but 70 ml. of thiosulphate added. If advisable, 0.5 ml. of N/10 iodine may be added to each flask and the titration repeated.

$$\text{Iodine value} = \frac{0.7 (\text{ml. used for blank} - \text{ml. used for oil})}{\text{Weight of oil taken}}.$$

The solution may also be made directly from iodine chloride, the strength being about 17 w/v. For other papers on the method, see Wijs (*Analyst*, 1899, **24**, 94 ; 1900, **25**, 31 ; 1902, **27**, 255), Lewkowitsch (*S.P.A.*, 1899, **24**, 257).

The increase in weight of oil when exposed to bromine vapour has been advocated as better than the Wijs method by Toms (*S.P.A.*, 1928, **53**, 71) and Croxford (*S.P.A.*, 1929, **54**, 445). For unsaponifiable matter Bolton and Williams have preferred to use the pyridine sulphate bromine reagent (*S.P.A.*, 1930, **55**, 6). For this method, see Rosenmund and Kuhnhehn (*Analyst*, 1924, **49**, 105, 400 ; 1932, **57**, 182).

CHAPTER XVIII

BUTTER, MARGARINE, CREAM

Keeping. Preservatives. Proximate analysis. Calculation of the composition of the fat. Birmingham prosecutions. Butter, effect of feeding, etc., adulteration, prosecutions. Bread and butter. Biscuits and butter. Margarine, adulteration, butter mixtures. Malt, banana, and nut cream butter. Cream, preservatives, thickening substances, analysis, prosecutions. Cream buns, sandwiches, trifle and eclairs. Clotted cream. Artificial cream.

BUTTER. MARGARINE

Two Committees have been appointed to make enquiries into the adulteration of butter and its prevention, and lengthy blue-books have been published which contain numerous analyses. The Interim Report of the Butter Regulations Committee (1902) recommended a limit of 16 % of water in butter. The Sale of Butter Regulations, 1902, raised the presumption, until the contrary is proved, that butter exceeding that limit is not genuine. The Final Report, 1903, recommended that a Reichert-Wollny figure of 24 should be a presumptive limit, and also that all margarine should contain 10 % of sesame oil, but no action followed these recommendations. The Butter Trade Committee in 1906 made a number of recommendations in relation to inspection, registration and labelling, most of which were included in the 1907 Butter and Margarine Act. Following this Act, the 1928 Act prohibits the importation of margarine containing more than 16 % of water, and declares that the presence of such margarine in a margarine factory is an offence (sect. 11 (1), 12 (f)). There is, however, no definite prohibition of the *sale* of such margarine.

WATER. That there is nothing unreasonable in these limits is shown by the following Birmingham figures :—

WATER IN BUTTER (3,283 samples) AND MARGARINE (3,917 samples)

PERCENTAGE OF WATER. Under 12 12— 14— 16— 18— Total

PERCENTAGE OF

SAMPLES.

Butter, 1921-23 .	36.6	35.4	25.6	1.9	0.5	100
„ 1924-30 .	23.4	44.6	30.1	1.3	0.6	100
Margarine, 1921-30	29.6	47.4	22.3	0.7	0	100

It is obvious that only a small proportion of the samples exceed the legal limit of 16 %, but comparison of the butter for the two periods indicates a marked decrease (36.3 % to 23.4 %) in the proportion of samples under 12 % of water, and an increase of those 14 % and over. It appears probable that the “ blending ” of butter has included the incorporation of water. If the water in butter

be raised from 11 % to 15 %, each £100 worth of it will yield £4 extra profit for water sold as butter. This is quite legal, but a degradation of the food value of butter. Excess of water in margarine is in a worse position, for while excess in butter *may* be natural water left in, excess in margarine has been intentionally added.

In 1902 four lots of cream were churned and salted under different temperatures and conditions at the British Dairy Institute; the water in the resultant butters only varied 11.4–11.6 % (*B.F.J.*, 1903, 98).

KEEPING. Three samples of butter were kept in parchment paper for ten months. The Reichert values of the fats fell from 0.2–0.6, the B.-R. readings increased by 0–0.7, while the average fall in Valenta value was 6°. The changes in three margarines were small and less constant, some giving increases and some decreases, in these values. Nine samples of the clarified fats of butter and margarine kept in open beakers for about three months were practically unaltered. The maximum difference in Valenta values was 2°, and the maximum difference in B.-R. 0.3.

Clayton has given the analysis of a sample of butter eighteen years old (*S.P.A.*, 1898, **23**, 36) and Shutt of two Canadian samples twenty years old (*S.P.A.*, 1925, **50**, 64). Five samples of butter six years old, examined by Arup (*S.P.A.*, 1929, **54**, 736) show a parallel between the increase of acidity and the decrease in the Reichert values. Other determinations were made. Elsdon, Taylor and Smith have given numerous analyses of rancid butters and margarines (*S.P.A.*, 1931, **56**, 515).

PRESERVATIVES. Birmingham butters were first tested for boric acid in 1896, and a conviction for the presence of 70 grains per lb. was obtained in 1898. At one time Alfred Hill condemned all samples of butter which contained boric acid, but that action was in advance of the time. The Preservative Committee of 1901 received evidence that 0.25 % of boric acid was sufficient and suggested a limit of 0.5 % to allow for errors in mixing. There were other prosecutions till 1904, which resulted in a great improvement, as is shown by the table below. The use of boric acid in margarine became illegal in 1927, and in butter in 1928 :—

BORIC ACID IN BUTTER AND MARGARINE

BORIC ACID ABSENT.		1899–	1906–	1916–	1926	1927	1928–30
Butter, percentage of samples		69	69	68	91	83	100
Margarine „ „		3	2	10	50	100	99.9
BORIC ACID PRESENT.							
Percentage of such samples containing 15 grains per lb. and over.							
Butter		54	39	17	14	2	—
Margarine . . .		50	47	20	26	—	0

PROXIMATE ANALYSIS. Proximate analyses of series of samples from forty dairies are given in Appendix XXX. of the Butter Regulations Committee, and Appendix 14 of the Report on Butter Trade Committee gives 101 analyses of butter and butter substitutes.

If a sample of butter appears wet, or has globules of water, or is likely to lead to a certificate for excess water, uniformity of the samples should be secured by transferring it to a wide-mouth glass-stoppered bottle, warming till the butter melts, then cooling with shaking till it sets.

Water is readily determined by weighing about 2 gm. in a metal dish 3 inches in diameter, with a flat bottom, and heating on the water-bath for an hour, and a subsequent half-hour to ascertain if there is further loss. A dry ashless filter should then be weighed with the dish and the fat extracted with petroleum ether and filtered through the paper; after drying, the residue is curd with salt, milk ash, and possibly boric preservative. After treatment with water and filtering, chlorine and boric acid may be determined in the filtrate. If the curd, etc., residue be ignored, salt, boric acid, and possibly water belonging to borax, may be lost and wrongly included in the curd. The fat may be weighed, or obtained by difference.

Salt may be determined in the butter itself by adding 10 ml. of boiling water to 3 gm. in a flask, shaking, and titration with N/10 AgNO_3 , and chromate (Arup, *S.P.A.*, 1929, **54**, 658), or by adding 10 ml. of acetone, in place of water, and titrating as before (Steuart, *S.P.A.*, 1928, **53**, 212), but see Davies (*S.P.A.*, 1932, **57**, 79).

If the presence of milk powder be suspected a rough test may be made for excess of lactose. Wash 5 gm. of butter with about an equal weight of boiling water, pour off the melted fat, add 3.5 ml. of Fehling solution to the aqueous residue, and heat in a test tube placed in a water-bath for ten minutes. A genuine butter will remain blue.

CALCULATION OF THE COMPOSITION OF THE FAT. The following formulæ have been used to determine the percentage of foreign fat in butter :—

$$\begin{array}{r} \frac{(5\text{—Soluble acids})}{5.0\text{—}0.5} \frac{100}{(225\text{—Saponification value})}, \frac{(Insoluble acids\text{—}89)}{95\text{—}89} \frac{100}{225\text{—}198}, \\ \frac{(.8663\text{—Sp. gr. } 99/15.5^\circ)}{.8663\text{—}.8610} \frac{100}{(26.0\text{—Reichert})}, \frac{(Iodine\text{ value—}35)}{70\text{—}35} \frac{100}{26.0\text{—}1.0} \end{array}$$

As an example, the analytical figures of a sample of "butter" are here given, with the amount of foreign fat found shown in

brackets :—Soluble acids 4.2 (18 %) ; Insoluble acids 90.3 (22 %) ; Saponification value 219 (22 %) ; Sp. gr. 0.8650 (24 %) ; Iodine value 43 (23 %) ; Reichert 20.5 (22 %). The sample was certified as adulterated with 20 % of foreign fat, and the vendor was fined.

As there is a natural variation in the composition of butter, and as margarine is made of various materials, such formulæ are based on averages, and identical figures of adulteration by various methods cannot be expected. In the presence of coconut oil, as indicated above, they are incorrect.

For the calculation of the percentage of butter in margarine the Committee of Analysts (*S.P.A.*, 1900, **25**, 310) gave figures depending on the formula :—

$$\text{Butter-fat \%} = \frac{(\text{Reichert}-0.9) 100}{32.0-0.9}.$$

As a high figure (32) is taken, the minimum amount of butter-fat is indicated, so that a vendor shall not be wrongly charged with excess of butter-fat in his margarine. An addendum to the Report (*S.P.A.*, 1909, **34**, 514) stated this formula must not be used if the figure for “insoluble volatile acids” (Polenske) exceeds 1, when coconut oil is probably present.

With illegal mixtures the above formula will underestimate the proportion of butter, and the following is better :—

$$\text{Average butter-fat \%} = \frac{(\text{Reichert}-1.0) 100}{29-1}.$$

In the presence of coconut oil or palm-kernel oil, the butter-fat is determined from the Kirschner and the Polenske by the formula given by Bolton, Richmond and Revis (*S.P.A.*, 1912, **37**, 185) :—

$$\text{Butter-fat \%} = \frac{\text{Kirschner}-(0.1 \text{ Polenske})-0.24}{0.244}.$$

For the detection of coconut oil in butter, the same writers (*opus cit.*) and Cranfield (*S.P.A.*, 1915, **40**, 439) have published analyses, and pointed out the importance of the relation between the Kirschner and Reichert figures. From these results Richmond (*S.P.A.*, 1919, **44**, 166) considers it may be safely assumed that if the Polenske is greater than 0.26 (Kirschner-10) the presence of coconut oil is established. He also gives a formula for the relation between the Reichert and Polenske. (See also Revis and Bolton, *S.P.A.*, 1911, **36**, 333.)

Many papers have been written on the above subjects and on the discrimination between coconut oil and palm-kernel oil in mixtures, which is much less important, from an adulteration point of view. The following authors may be mentioned :—Shrewsbury and Knapp (*S.P.A.*, 1910, **35**, 385 ; see also *S.P.A.*, 1911, **36**, 195, 334, 448 ; 1917, **42**, 72, 295), Cribb and Richards (*S.P.A.*, 1911, **36**, 327),

Arnaud and Hawley (*S.P.A.*, 1912, **37**, 122), Bolton, Richmond and Revis (*S.P.A.*, 1912, **37**, 183), Gilmour (*S.P.A.*, 1920, **45**, 2; 1921, **46**, 183; 1925, **50**, 119, 272), Elsdon and Smith (*S.P.A.*, 1925, **50**, 53; 1927, **52**, 63, 317), Cocks and Nightingale (*S.P.A.*, 1928, **53**, 322).

BIRMINGHAM PROSECUTIONS FOR BUTTER AND MARGARINE. 1874. Water 19·1 %, salt 9·9 %, foreign fat. Fine 5s.

1896. Margarine unlabelled when exposed for sale. Fine £10.

1898. Boric acid 70 grains per lb. Expert evidence was given. Fine £1 (*F. & S.*, 1898, May 21).

1898. Foreign fat 20 %, boric acid 21 grains per lb. Fine £20. An appeal to Quarter Sessions was dismissed, the Recorder finding that the penalty was not too severe, and that "Finest pure butter, guaranteed," branded on the lid of the butter tub, was not a legal written warranty.

1898. Wholesale consignment of unmarked margarine. The manufacturer was fined £20 for each of two offences, and also £2 for having an unregistered margarine factory (*F. & S.*, 1898, Dec. 3).

1901. Water 22 %, being at least 6 % in excess, boric acid 1 %. Two other analysts found 1·03 % and 1·08 % of boric acid. The Government analysts found 21·25 % of water and 0·39 % of boric acid. Fine £5. The article was milk-blended butter. The samples were not put in bottles, but in parchment paper (*B.F.J.*, 1901, 200).

1901. Water 19·5 %, boric acid 1·1 %. The case against the vendor was dismissed as he proved a warranty. The wholesale dealer was fined £10 for giving a false warranty.

1901. Foreign fat 87 %. It was labelled "Pure Danish butter," but that was only true for a small piece carefully fitted into a large lump of margarine. The assistant had been told to serve the butter part to suspicious customers only. Fine £20.

1901. Retail sale not marked "Margarine." The case against the owners was dismissed, but they prosecuted the assistant, and he was fined £2.

1902. Wholesale consignment of unmarked margarine. The sample was taken in course of delivery to a small shop, and the wholesale dealer was fined £50. He had supplied two other small shops with margarine as butter.

1903. Water 31 %. Fine £5. The sample did not look wet.

1906. Retail sale not marked "Margarine." The vendor dressed as a countrywoman and carried the butter and eggs in a farmer's basket; she occasionally asked for orders for pork from her "own pigs." The margarine had been bought at 6½d. per lb., and after making up as farmer's butter in a cellar, had been sold at

1s. 2d. per lb. She was fined £10, but did not pay it, and went to prison for two months with hard labour.

1906. Applying false trade description "Danish butter," and falsely applying the Danish "Lur" brand, to margarine supplied to the City Hospital. One of the defendant's witnesses confessed that he obtained empty Danish butter tubs, and packed margarine in them. Fine £20 and £30 costs under the Merchandise Marks Act (*Grocer*, 1906, March 3; *B.F.J.*, 1906, 48).

1906. Retail sale not marked "Margarine." It was alleged that a mistake had been made, but rebutting evidence was given that four out of five previous informal samples were margarine. Fine £10.

1907. Wholesale consignment not marked "Margarine." It was taken from a 56-lb. box at a railway station in course of transit to a creamery. It was part of a consignment of 5 cwt. of "butter." Fine £20.

1907. Butter in excess 40 %. It was proved that in the previous four months twenty-seven samples of "butter" had been bought at the shop, and that ten of them were illegal mixtures of butter and margarine. Fines £11, including £1 for the retail sale of unmarked margarine (*Grocer*, 1907, Aug. 17).

1925. "Boric preservative equivalent to 40 grains of boric acid per lb. Observations. "Butter should not contain more boric preservative than is equivalent to 21 grains of boric acid per pound." The Government analysts found 40.6 grains. The objection to the form of the certificate was overruled by the magistrates. Fine 10s. (*B.F.J.*, 1925, 46).

BUTTER

EFFECTS OF FEEDING, ETC. Appendix XXIX. to the Report of the Butter Regulations Committee is a report by T. E. Thorpe on the composition of 357 samples of British butter from forty herds in widely spread districts. The majority of the samples from each dairy gave Reichert figures above 24, but some samples from eight dairies, usually taken in November or December, were below this figure. Four of these were small (three to nine cows), and the lowest figure was 19.4. He concluded that feeding cows on grass alone increased the Reichert figure of their butter, but that when autumn grass formed the main food of the cows, there was a tendency for that figure to decrease. As the lactation period proceeded, there was a decrease in the Reichert figure. Determinations of the sp. gr. at 100° F., the Koettstorfer number, and the B.-R. figure were also made on the samples (see also *Analyst*, 1904, 29, 113). Butter made by Smetham from the milk of cows advanced in lactation gave a Reichert figure of 17.9 only (*S.P.A.*, 1909, 34, 304).

Earlier experiments by T. E. Thorpe (*Analyst*, 1898, **23**, 255) proved that cows fed on cottonseed cake might yield butter giving a reaction for cottonseed oil, not exceeding that given by 1 %, but that feeding with sesame oilcake could not produce a reaction for that oil in the butter.

A number of feeding experiments made at the Midland Agricultural and Dairy College have been recorded by Cranfield (*S.P.A.*, 1911, **36**, 445 ; 1915, **40**, 433 ; 1916, **41**, 336). Channon, Drummond and Golding have given detailed results of feeding experiments on three individual cows (*S.P.A.*, 1924, **49**, 311). Brownlee (*Scient. Proc. Royal Dublin Society*, 1925, 49) has shown that a large proportion of Irish winter butters (October to January) give a Reichert-Wollny figure below 24, the lowest being 19.5 (see also *Analyst*, 1910, **35**, 381). Arup has published a study of Irish winter butter (*S.P.A.*, 1929, **54**, 634).

ADULTERATION OF BUTTER. This has passed through a variety of phases, and has necessitated the passing of Acts of Parliament, which generally have been successful in accomplishing their purpose.

The first artificial butter was the result of the experiments of a French chemist, Mège Mouriès, and came into commerce about 1872, its production having been stimulated by the shortness of butter during the siege of Paris.

In the decade commencing 1878 about one-third of the Birmingham samples of butter were adulterated with foreign fat. When exposed for sale it was sometimes marked "Butterine," with the tickets so arranged that the last three letters were not visible. Owing to the resemblance of the words, conflicting evidence was often given as to whether "butter" or "butterine" was requested, and the Margarine Act required that at all stages the article should be marked "Margarine." Before 1885 the substitute was margarine itself, but in 1887 mixtures containing one-third to two-thirds of butter were common. These were intended to be sold as butter, and not as a mixture. The Act of 1899 was a unique law to prevent the quality of margarine being too good. It prohibited the sale, etc., of margarine "the fat of which contains more than 10 % of fat derived from milk." This corresponds to about 9 % of butter-fat in the margarine. The Act inconsistently allowed the importation of margarine containing "10 % of fat derived from milk." This anomaly remains in the 1928 Act (sects. 6 (2) and 12 (e)).

About 1902 an enterprising firm commenced selling a blend of butter and milk as "butter." It had an agreeable taste, was more easily spread than butter, and was sold at a lower price, but as it contained 20-30 % of water, the price was sufficiently high to yield a good profit. Much litigation ensued, and the 1907 Act required

the article to be sold as "milk-blended butter" with not more than 24 % of water.

The variation in the percentage of adulteration in England and Wales and also in London is given below. As in Birmingham a number of adulterated samples were frequently bought from one shop, percentages of adulteration would be misleading, so comparative adulteration figures (see p. 114) are given instead :—

ADULTERATION OF BUTTER

Period.	1878—	1888—	1898—	1908—	1918—	1928—30
PERCENTAGE OF ADULTERATION.						
England and Wales	. 16·8	11·1	6·8	5·7	2·0	1·7
London	. 16·7	8·7	10·4	8·3	2·1	2·4
COMPARATIVE ADULTERATION						
FIGURE—Birmingham.						
Foreign fat	. 34·1	14·4	5·1	1·1	0	0
Excess water.	. 0	0·3	0·8	0·1	0·9	1·4

It will be seen that in Birmingham, though adulteration with foreign fat has ceased, adulteration with water has increased. The war made a great difference to the estimation in which margarine was held. Before it some people were ashamed to ask for "margarine" and used a synonym, such as "mild butter."

USUAL VALUES OF CONSTANTS OF BUTTER AND MARGARINE

Constants.	Butter.	Margarine.
Soluble acids (as butyric acid). 1878—89	4·8—5·8	0·1—0·8
Insoluble acids (Hegnner number) ,,	88·0—90·5	93·5—96·0
Saponification value	218—232	196—201
Sp. gr. 99/15·5° (1888—97)	0·8658—0·8670	0·8600—0·8620
Valenta number ° C. (1890—3)	58—68	100—110
B.-R. 40° (1926—7)	42—45	41—52

With the first five constants given there is a fair distinction between butter and margarine. These, however, were all determined before 1906, about which date coconut oil was first commonly used as a constituent of margarine. The constants of this oil have the unfortunate analytical property of being on the other side of butter to the older margarine (cp. Muter, *S.P.A.*, 1891, **16**, 88). For example, the saponification value of it is about 260, and therefore a mixture of it with margarine may give the value for genuine butter. The B.-R. of the 1926—7 margarines (41—52) include the butter range (42—45), and therefore this constant is of little or no value for detecting a coconut oil margarine.

Comparison of the following figures obtained with samples of Birmingham butter and margarine, will show that the

above-mentioned disadvantage does not apply to the Reichert value :—

REICHERT VALUES OF BUTTER (3,607 samples) AND MARGARINE (1,071 samples)

	REICHERT VALUES	20-	22-	24-	26	28	30-	32-	Total.	
	Butter, 1920-30	.	.	0.7	8.5	27.7	38.3	19.9	4.9	100.0
*	First Quarters	.	0	0.3	4.6	23.3	37.8	25.8	8.2	100.0
	Second „	.	0	0.2	3.9	18.8	40.0	29.4	7.7	100.0
	Third „	.	0	0.1	6.7	31.9	47.0	14.1	0.2	100.0
	Fourth „	.	0.1	2.1	19.0	37.5	27.8	9.9	3.6	100.0
	REICHERT VALUES	0.8-	2-	3-	4-	5	6-7		Total.	
	Margarine, 1928-9	.	5.2	10.7	18.7	27.0	23.6	14.8		100.0

As the Reichert value of coconut oil is about 8, there is a considerable difference between it and butter.

It should be noted that the proportion of samples of butter giving Reichert figures less than 24 is very small and almost confined to the fourth quarters, also that there are a much larger proportion of samples 30 and over in the first two quarters than in the last two. This is an indication of the seasonal variation previously mentioned.

PROSECUTIONS FOR WATER, MILK-BLENDED BUTTER.

Manchester. Water 26.8 % instead of 15 %. It was Irish butter, and the wholesale dealer was fined £1 for false warranty. The hearing of several similar cases occupied three days (*F. & S.*, 1894, Jan. 13, 20 ; Feb. 3, 10).

Ramsgate. Added water 5 %, the total amount being 21.1 %. Dismissed : the magistrates held that the excess water was due to the preparation, but was not “added water” (*F. & S.*, 1896, Nov. 14).

Longton, Brierley Hill. Water 22.5 %, being at least 6 % too much ; 21.75 %, being at least 5 % too much. The vendors admitted the addition of milk to the butter, and the fine was £20 in each case. On appeal, *Pearks, Gunston and Tee v. Van Tromp*, and *Same v. Knight*, the convictions were confirmed. It was held that the watery part of the added milk, instead of being eliminated, was left in considerable quantities in the product, which was therefore the genuine article saturated with a spurious article. This was fraudulently done, but motive was immaterial for a conviction (*B.F.J.*, 1901, 266).

Pearks, Gunston, and Tee v. Houghton. An appeal from a fine of £20 for selling butter containing 23.8 % of water. The conviction was quashed ; an inner wrapper stating admixture was not considered sufficient, but in addition there was a large notice in the shop (*B.F.J.*, 1902, 56 ; *Analyst*, 1902, 27, 110).

Hayes v. Rule and Law. The Court held that the sale of a butter

containing 20.6 % of water was legal if the article was wrapped in a notice "disclosing the fact that the article was a mixture" (*B.F.J.*, 1902, 109; *Analyst*, 1902, 27, 165).

Bayley v. Pearks, Gunston, and Tee. The Court decided that milk-blended butter was not "margarine, the fat of which contained more than 10 % of butter fat" (*B.F.J.*, 1902, 107; *Analyst*, 1902, 27, 167).

Bristol. Milk 9 %, added water 28 %, the total amount of water being 46.7 %. A second sample had 50.8 % of water. Fine 4 3 guineas in each case (*Grocer*, 1904, Sept. 3).

Harlesden. Water 30.5 %. The wrapper stated that it contained about 24 % of water, but that amount was not guaranteed. The Government analysts found 31.7 %. Fine £20 (*B.F.J.*, 1906, 73).

London, Stratford. Water 18 %. Fine £2 (*Grocer*, 1930, Dec. 13).

Belfast. Moisture 27.4 %. Fine 5s. (*Grocer*, 1931, Nov. 14).

PROSECUTIONS FOR FOREIGN FAT. *Myers v. Gregory* was an appeal case in which the High Court decided that if a person asked for butter, he ought not to be given something which was not butter (*B.F.J.*, 1906, 29).

Manchester. False trade description of "butter" which contained not less than 10 % of coconut oil. At the eighth day's hearing, the stipendiary said that, although the butter was not pure, he did not think the defendants had intended to defraud, and dismissed the case (*Grocer*, 1906, March 17, 24, 31; May 5, 19; June 30).

Bournemouth. Water 27.3 %, being 11 % in excess, and that approximately 7 % of the fat was coconut oil. The Government analysts reported approximately 5 % of coconut oil in the fat. Fine £1 and £9 costs, and the same amount in a similar case (*B.F.J.*, 1907, 99, 194).

London, Tower Bridge. Margarine 40 %. The vendor had made "tenpenny butter" by mixing shilling butter with eightpenny margarine. Fine £3 (*Grocer*, 1907, June 8; *B.F.J.*, 1907, 119).

Clerkenwell. Too high a percentage of butter. The purchaser asked for "1s. salt." The magistrate considered the term ambiguous, and dismissed the case (*Grocer*, 1911, April 29).

Eastbourne. Fat, other than butter-fat, at least 80 %. The defendant had issued circulars offering "butter of excellent quality." A postal order was sent to London, and the sample was received. Fine £10. A fine of £10 was also inflicted at *Bradford* for a similar offence (*B.F.J.*, 1912, 73).

North London. Foreign fat 40 %. Another analyst found 41 % in the same sample, but the other two samples were found to be genuine butter. Dismissed (*B.F.J.*, 1916, 293).

Listowel. Fat other than butter-fat 13 %. The Reichert figure was stated to be 22.1. Three analysts were said to have obtained Polenske figures of 3.1, 1.9, and 1.6. Fine £1 1s., which was reversed by the Circuit Court (*Grocer*, 1927, Feb. 19 ; May 21).

Llandilo. Margarine 20 %. The Government analyst and another analyst (whose Reichert figure was 24.5) found no foreign fat. Dismissed (*Grocer*, 1929, Feb. 23 ; *B.F.J.*, 1929, 35).

London, Old Street. Foreign fat of vegetable origin not less than 10 %. The butter was said to have been sold as imported. Fine £20 and £25 costs. The defendant was the occupier of a margarine factory next door, and had been previously fined (*Grocer*, 1929, May 11 ; *B.F.J.*, 1929, 64).

London, Tower Bridge. Foreign fat 20 %. Other samples had been of similar composition. Fine £20 (*Grocer*, 1930, Nov. 15 ; *B.F.J.*, 1930, 116).

PROSECUTIONS FOR VARIOUS OFFENCES IN RELATION TO BUTTER. *Folkestone.* Boracic acid 25 grains per lb. Fine £50. On appeal to Quarter Sessions the conviction was quashed (*B.F.J.*, 1901, 328, 353).

London, Bow Street. Excess water 1.48 %, starch 0.53 %. Fine £2 (*Grocer*, 1907, Jan. 19 ; *B.F.J.*, 1907, 28).

Wolverhampton. Selling a mixture of condensed milk and butter to which a false trade description "guaranteed pure butter" had been applied. A Government analyst found proteids 1.19 %, lactose 1.23 %, and preservatives 1.24 %. For two offences the defendants paid £40 10s. in fines and costs (*Grocer*, 1907, Nov. 2 ; *B.F.J.*, 1907, 195).

London, South-Western. Having on their premises a mixture of milk powder and butter intended for admixture with butter. A Government analyst had found in the defendant's butter, curd 2.29 % (which contained lactose 1 %) and albuminoids 1.06 %. As a rule genuine butter contained less than 0.3 % of lactose, and the maximum percentage was 0.4. The magistrate was satisfied that about 1 % of milk powder was used in the blending of the butter, and that the admixture constituted adulteration. Fine £5 and 80 guineas costs (*Grocer*, 1912, March 2, 23 ; *B.F.J.*, 1912, 47, 49).

London, Tower Bridge. Sodium fluoride 0.21 %. Medical evidence was given that fluorides ought never to be used as food preservatives. They accumulated in the body and produced nausea and diarrhoea. Costs 10 guineas (*Grocer*, 1913, March 22 ; *B.F.J.*, 1913, 50).

Liverpool. Cane or beet sugar 1.2 %. The stipendiary thought the sugar was used as a preservative, and that its presence was not an infringement of the Act (*Grocer*, 1913, Sept. 27).

Nottingham. Obtaining money by false pretences by selling

margarine as "best English butter." In ten days he had sold 241 lb. of margarine, buying it at 6d. lb. and selling it at 1s. Previous convictions were proved and the defendant was sentenced to three years' penal servitude (*Grocer*, 1914, Jan. 10).

Taunton. Applying false trade description "Guaranteed pure butter" to an article which contained 3 % added milk powder. The total of the fines and costs was £92 10s. (*Grocer*, 1915, Jan. 16 ; *B.F.J.*, 1915, 12).

New Ross. Salt 12·8 %, the normal standard being 4 %. Fine £1 (*Grocer*, 1924, April 12).

London, Tower Bridge. Having margarine on premises intended to be used for adulterating butter. The defendant pleaded ignorance of the fact that there was 35 % of foreign fat in the adulterated imported butter, and therefore they intended blending but not adulteration. The magistrate accepted this statement, and ordered him to pay 10 guineas costs (*Grocer*, 1929, Jan. 12 ; *B.F.J.*, 1929, 15).

Magherafelt. Boric acid 0·075 %. Two vendors, who bought their butter from a local creamery, were each fined 5s. (*Grocer*, 1929, June 8).

London, Tower Bridge. Boric acid 0·14 % and 0·16 %, respectively. The summonses were withdrawn, as the Authority was satisfied that the defendants were not responsible for the adulteration (*Grocer*, 1930, Nov. 22).

Kensington. Boron trioxide equivalent to 13·881 grains of boric acid per lb. Fine £2 (*Grocer*, 1930, Dec. 20).

London, Old Street. Free fatty acids 10·52 %, being 8·5 % in excess. The butter was rancid. Fine 1s. (*Analyst*, 1930, 55, 633).

PROSECUTIONS FOR BREAD AND BUTTER. *Birmingham*. Fat 5 %, of which 90 % was foreign to butter. Fat 11 % entirely foreign to butter. Each vendor was fined 5s., as were eight others for similar offences (*B.F.J.*, 1900, 105).

London, Mansion House. Bread and margarine 100 %. The magistrate was of opinion that the certificate was bad, and he dismissed the case because each sample consisted of one slice, while each slice should have been divided (*B.F.J.*, 1903, 114).

Birmingham. Margarine nine parts, bread ninety-one parts. Margarine fifteen parts, bread eighty-five parts. Each vendor was fined £2 (1903 Report).

Darlington. The fat contained 47 % of fat other than butter fat. Fine £10 (*B.F.J.*, 1930, 117).

PROSECUTION FOR BISCUITS AND BUTTER. *Liverpool*. The "butter" was margarine. It was sold in a vegetarian restaurant, and the owner said his customers expected nut butter. Fine £5 (*B.F.J.*, 1912, 31).

MARGARINE

ADULTERATION. BUTTER MIXTURES. Of the samples of margarine examined in England and Wales during 1902–13, 3·9 % were adulterated, but during 1919–30 the proportion fell to 1·0 %. The adulterants included preservatives, excess of butter and water. Labelling offences, examples of which are given below (p. 259), are not included.

The Report on Food Products Adulteration, issued in 1896, recommended the prohibition of the sale of mixtures of butter and margarine. Manufacturers pleaded that as milk was used in the manufacture of margarine, butter might be present in the margarine up to 5 % ; to be on the safe side 10 % was allowed in the 1899 Act, but there was no thought that butter would be intentionally added.

ANALYSES OF MARGARINES AND MIXTURES

Description.	Reichert.	Polenske.	Kirschner.	B.-R. 40° C.	Valenta °C.
1. "Margarine" . . .	0·7	1·4	—	48	108
2. " " . . .	8·0	11·4	—	38	36
3. " " . . .	3·6	4·6	—	46½	94
4. "Butter" . . .	10·1	1·5	—	46	87
5. "Mixture of pure Irish butter and choice margarine" . . .	4·6	2·3	2·9	47½	89
6. "Margarine mixed with butter" . . .	4·5	4·7	0·9	45	—
7. "Contains 6 % butter" . . .	9·1	9·5	3·5	—	—

No. 1 is an example of "oleo margarine" which was defined in Food Order No. 1,162, of 1917, as containing "not less than 55 % of the following fats :—oleo oil, oleo stearine, premier jus, and choice neutral oil, but no hardened oil." No. 2 had about 70 % of coconut oil, and No. 3 about 50 % of palm-kernel oil. No. 4, though sold as butter, contained only about 30 % of average butter, and the vendor was fined £20. From the description of No. 5 a purchaser would expect more than the 10 % of butter actually present, while No. 6 had a mere trace of butter. In No. 7, perhaps through bad mixing, 9 % of butter was present.

Henville and Paulley have found that most margarines contain a dye which is absent from butter. This fact may sometimes be useful for detecting a mixture containing 10 % of margarine (*S.P.A.*, 1929, 54, 413).

PROSECUTIONS FOR MARGARINE. *Clerkenwell.* Paraffin wax 6·7 %. Fine and costs 47s. (*F. & S.*, 1898, Jan. 29).

Rushden. "Water 21 % (an excess of at least 5 % on the amount of water which margarine should contain)." The magistrates found that the amount of water present was excessive ; that the article was adulterated with water, and fined the vendor £1.

On appeal, *Burton & Sons v. Mattinson*, the conviction was confirmed, the Court finding that the article was margarine and water, and not margarine (*B.F.J.*, 1902, 110 ; *Analyst*, 1902, **27**, 169).

Londonderry. Exposure for retail sale of a parcel of margarine without having it properly labelled. The margarine was in an open butt. The defence argued that the branding on the butt was sufficient labelling and that it was a package and not a parcel. The case was dismissed, but on appeal to the King's Bench Division, Dublin, *Maguire v. Porter*, the Court held that the magistrates should have convicted, as in the case the package became a parcel within the meaning of the section (*Grocer*, 1904, Nov. 12).

Darwen. Exposure for retail sale of unmarked margarine. A pyramid of six pound packets of margarine in a shop window had only one label. The magistrates held that the six packets were a "parcel" and that one label was sufficient. Their decision was confirmed on appeal, *Parkinson v. M'Nair* (*B.F.J.*, 1905, 155).

Bolton. Margarine containing not more than 75 % of fat, and excessive amounts (5.5 %) of curd, sugar and starch. The average amount of fat in margarine was given as 85 %. Fine £5 and £30 costs. On appeal—*Roberts v. Leeming*—the conviction was confirmed. The Court held that, as there was no statutory standard for margarine, the magistrates must themselves fix a standard, and that there was sufficient evidence for them to conclude that margarine, as usually sold, contained at least 85 % of fat (*B.F.J.*, 1905, 155).

Wolverhampton. Selling margarine by a name other than that of margarine, viz., "Keeloma." The certificate, which stated the sample was "margarine, containing only 3 % of butter," was held by the magistrate to be insufficient, as it did not enable him to decide if an offence had been committed. On appeal, *Alhwood v. Gregory*, the Lord Chief Justice held the certificate showed that the sample was margarine, and that it did not contain more than 10 % of butter-fat, and was sufficient. On rehearing, the defendant was fined £5 and £10 special costs, as well as 10s. for each of four other offences (*B.F.J.*, 1905, 177, 225).

North London. Water 29.4 %, mashed potatoes 13 %. The imported margarine was said to have been intended for Germany, where such mixtures were allowed. Fine £10 (*Grocer*, 1906, Dec. 8 ; *B.F.J.*, 1907, 12).

Royton. Water 24.7 %, commercial glucose 4 %, which had been put in to prevent the water exuding. Fine £10 (*Grocer*, 1907, March 16 ; *B.F.J.*, 1907, 69).

Derby. Retail sale of unmarked margarine. "Margarine 100 parts, butter none." The defendant stated that the article was prepared entirely from coconuts, and was not margarine—which contained animal fats. The case was dismissed, but the appeal,

Wilkinson v. Alton, was allowed. The Court held that everything prepared to look like butter, which was not butter, must be called margarine (*Grocer*, 1908, April 11 ; *B.F.J.*, 1908, 9, 61, 84).

Wolverhampton. Advertising margarine by another name than margarine, viz., "our shilling" in a placard "Please ask to taste our shilling." Fine and costs 56s. (*Grocer*, 1909, Jan. 9, 23 ; *B.F.J.*, 1909, 28).

Glasgow. Soft paraffin 16 %. It was said to have been sold as imported, and the Sheriff dismissed the case (*Grocer*, 1909, Dec. 4 ; *B.F.J.*, 1910, 11).

Leeds. Selling margarine in a wrapper on which the word "Margarine" was not printed in capital block letters so as to be distinctly visible. Witnesses connected with the printing trade agreed that the epithet "block," as applied to letters, had no significance. The letters were green on the inside of a semi-transparent wrapper. Fine £3 (*Grocer*, 1912, Nov. 30 ; *B.F.J.*, 1912, 233).

Salford. Importing margarine containing 16.8 % and 16.7 % of moisture. Fine £50 (*Grocer*, 1921, Feb. 19 ; *B.F.J.*, 1921, 27).

London, Thames. Mineral oil 9.5 %. Observation "Mineral oil is devoid of any food value." Fine £1 (*Analyst*, 1926, 51, 459).

Lambeth. Boric acid 0.31 %. Fine £20 (*B.F.J.*, 1929, 36 ; *Grocer*, 1929, Feb. 16).

Southampton. Water 17.4 %. It was pointed out that the legal limit of 16 % only applied to margarine as prepared for sale, and not as sold. The article had been bought with a warranty, but this did not apply as it had been beaten on the block. Fine 10s. (*Analyst*, 1929, 54, 232 ; *Grocer*, 1929, Feb. 9).

London, Marylebone. Sale in a wrapper not marked, "Margarine." The vendors had received the article in ½-lb. packets, and had each cut one in half for the sale, with the result they were only marked "Mar." Three vendors were each fined £1 (*Grocer*, 1929, Aug. 17).

North London. (1) Selling margarine in plain wrapper, which (2) contained 85 % of butter fat. (3) Exposing margarine for sale without a label. The magistrate believed the defendant had acted honestly, and ordered him to pay 2s. costs in each case (*Grocer*, 1929, Sept. 28 ; *B.F.J.*, 1929, 115).

London, Old Street. Consigning margarine containing 18.4 % of water. Ordered to pay 12 guineas costs (*B.F.J.*, 1930, 36).

Abercynon. At least 40 % of fat not derived from milk. Fine £2 (*Grocer*, 1931, Jan. 31 ; *B.F.J.*, 1931, 28).

PROSECUTIONS FOR BUTTER MIXTURES. *Marylebone*. "Butter mixture" containing 4½ % of butter. Fine £20. On appeal, *Anness v. Grivell*, the conviction was quashed. Although it was described as a "very good mixture," the Court felt the

purchaser was not prejudiced, as it could not legally contain more than 10 % of butter (*Grocer*, 1915, March 27).

Salford. "Margarine blended with butter" which contained only 0.5 % of butter. The summons against the vendor was dismissed and the manufacturer was fined £20 for aiding and abetting (*B.F.J.*, 1924, 16. A similar case is given *Analyst*, 1924, **49**, 476).

PROSECUTION FOR MALT BUTTER. *Glasgow*. Retail sale of margarine not marked "Margarine." Margarine fat 44.3 %, dry malt extract 39.8 %. The case was dismissed as the article did not resemble either butter or margarine closely enough to deceive a purchaser (*Grocer*, 1915, March ; *B.F.J.*, 1915, 50. A similar case is given *Grocer*, 1915, Oct. 2 ; *B.F.J.*, 1915, 192).

PROSECUTION FOR BANANA BUTTER. *Castle Eden*. It was labelled "Containing ripe bananas and butter," but the fat was vegetable, not butter fat. The defence argued that they were entitled to use the general term "butter," as no one was entitled to assume that it indicated only cow's butter. On the understanding that the labels were to be altered, the defendants were ordered to pay costs only (*Grocer*, 1920, Nov. 27 ; *B.F.J.*, 1920, 114).

PROSECUTION FOR NUT CREAM BUTTER. *Douglas*. Retail sale of unlabelled margarine. Fat, or coconut oil, 90.4 %, non-fatty solids 9.2 %, moisture 0.4 %, butter none. Fine £2 (*Analyst*, 1923, **48**, 325).

CREAM

When cream was obtained by hand-skimming milk which had stood, the proportion of fat in it was not likely to exceed 40 %, and would usually be much less. The introduction of the centrifugal separator made it possible to have cream much richer in fat, and to control the amount in it. There is no standard for the amount of fat in cream, the nearest approach being in the Milk and Cream Regulations of 1912, which prohibited the addition of preservative to cream containing less than 35 %. The Scotch Inter-departmental Committee of 1922 considered that "Double Cream" should contain at least 40 % of fat. Elsdon and Stubbs (*S.P.A.*, 1930, **55**, 125) have suggested that standards of 15 % of fat for "hand-skimmed cream," 30 % for "cream," and 45–50 % for "thick cream," are desirable. The Ministry of Health Report for 1922 states that "it is fairly generally accepted in this country that cream should contain 40–50 % of butter-fat, with about 5 % of non-fatty solids."

FAT IN CREAM AND PRESERVED CREAM, 1913–30 (458 samples)

Percentage of fat	.	.	20–	25–	30–	35–	40–	45–	50–	55–	60–	71	Total.
Percentage of samples	.	.	7	10	2	3	7	20	31	14	6		100

It will be seen that no less than 65 % of the samples contained 45–59 % of fat, while 17 % were about half that strength containing

20–29 % of fat. All but six of the eighty-six samples containing less than 35 % of fat were tinned sterilised cream. For practical purposes, it may be stated that fresh cream contains over 40 % of fat, and tinned cream about 23 %.

In 1927, 16·4 % of the samples of cream examined in England and Wales were reported adulterated, but in 1928–30 the proportion had fallen to 2·4 %.

It is unsatisfactory that the word “cream” should cover such a variety of richness, and the anomaly is made worse by tinned cream often being marked “Thick cream.” In such cases the thickness is due to sterilisation, and not to a good proportion of fat as in the old-fashioned thick cream. The use of the words “Pure Rich Cream” or “Highly Concentrated Rich Dairy Cream” for articles containing only 20–23 % of fat makes the labels false ones. The use by English dairymen of a label guaranteeing that their cream contained at least 40 % of fat would inform the public of its superiority over the tinned cream.

Although there is such a wide variation of the amount of fat in cream, the ratio of the solids-not-fat to the water is practically constant (Richmond, “Dairy Chemistry”; Leonard and Smith, *S.P.A.*, 1896, **21**, 283). The percentage of solids-not-fat in the non-fatty part of normal cream is about 8·7–9·5. It will be diminished by addition of water, and increased if there has been evaporation during sterilisation or the preparation of clotted cream : in the latter there may be 15 % or more. High solids-not-fat may be due to the addition of condensed milk (Richmond, *S.P.A.*, 1909, **34**, 210).

SOLIDS-NOT-FAT IN CREAM AND PRESERVED CREAM (119 samples)

Percentage of solids in non- fatty part .	3·7–	5·0–	6·0–	7·0–	8·0–	9·0–	10·0–	11·0–13·0	Total.
Percentage of samples .	12	12	13	18	19	10	9	7	100

These figures indicate that the reduction of the strength of cream by the addition of water is fairly common in Birmingham (see Lerrigo, *S.P.A.*, 1928, **53**, 488). The addition of water or colouring matter to cream was prohibited by the Milk (England and Wales) Order, 1921. Richmond (*S.P.A.*, 1914, **39**, 246) has advocated the determination of the aldehyde figure for the detection of water in cream.

PRESERVATIVES. The Milk and Cream Regulations of 1912 and 1917, requiring the labelling of preserved cream, produced a great diminution in the use of boric acid. Of the few samples of Birmingham cream examined 1896–1912, 83 % contained boric acid ; during the Regulations period, 1913–27, the proportion fell to 43 % ; and 72 % of the preserved creams were correctly labelled. After

the 1925 Regulations came into force, prohibiting its use, only 1.7 % of the samples examined 1928-30 contained boric acid. There was also a great fall in the amount used ; in the earlier period as much as 1.0 % of boric acid was found, while those of the last three years only contained about one-tenth of this amount. In 1909 Hamill made a Report (No. 10) to the L.G.B. on cream and the use of preservatives in it. It had an addendum by Monier-Williams on the detection of benzoic and salicylic acids and saccharin.

THICKENING SUBSTANCES. The Milk and Cream Regulations of 1912 prohibited the addition of thickening substances to cream and defined them as sucrate of lime, gelatin, starch paste, or other thickening substance, but permitted the use of sugar. The Regulations of 1925 repeated this definition. Viscogen, a solution of lime in syrup, has a marked thickening power. Pyne (*S.P.A.*, 1930, **55**, 747) uses the decrease of viscosity produced by adding potassium oxalate for detecting it. Stokes (*S.P.A.*, 1897, **22**, 320, also 322) and Mendelsohn (*S.P.A.*, 1930, **55**, 567) have given methods for detecting gelatin.

ANALYSIS. *Total solids* may be determined on about 2 gm. in a flat-bottom dish ; tilting the fat to one side on the water-bath promotes the drying ; the residue is dried in the water oven about six hours. Fuming, continual loss of weight, and browning indicate the presence of glycerin (Lerrigo, *S.P.A.*, 1928, **53**, 335).

Fat may be determined by the Gottlieb, Werner-Schmid or Adams method. In the latter case, about 2 gm. is weighed in a porcelain dish and diluted with about 2 ml. water before putting on the coil.

The Gerber method with the special tubes may also be used. About 5 gm. is weighed in the small cup, and added to 10 ml. of water, 10 ml. of the sulphuric acid, and 1 ml. of amyl alcohol in the butyrometer. The tube is shaken before and after putting in hot water (65° C.) until the fat separates. Gases may require to be liberated through the small opening. Centrifuge five minutes, read and repeat till constant reading. If exactly 5 gm. be taken the reading is the percentage of fat, otherwise it must be corrected. The volume occupied by 60 % should be 3.34 ml. Lampitt, Hughes and Bogod (*S.P.A.*, 1924, **49**, 418) consider results obtained by the above method are too high, and recommend the use of only 0.25 ml. of amylic alcohol, using the factor 0.111, and adding 0.0054 for the meniscus.

PROSECUTIONS FOR CREAM. *Dundee.* Skimmed milk at least 45 %. The sheriff dismissed the case as no adulteration had been proved and no evidence had been given that the article was not worth the money paid for it (*B.F.J.*, 1901, 61).

Blackpool. Boiled wheaten flour 2 %, and a little boric acid. Fine 1s. (*B.F.J.*, 1902, 116).

Glasgow. Importing cream not conspicuously marked that it contained gelatin. The sheriff dismissed the complaint as he could not presume that the presence of the foreign substance had injuriously affected the quality of the cream (*Grocer*, 1907, March 3).

Westminster. Boric acid 22 grains per lb. and injurious to health. Labelled "Rich cream, contains a small percentage of boron preservative to retard sourness." Fine £2. An appeal to Quarter Sessions was dismissed, as also one to King's Bench (*Cullen v. McNair*) (*B.F.J.*, 1907, 154; 1908, 97, 100, 151; *Grocer*, 1907, Nov. 9, 23).

Dundee. Adulterated with water 9 %. The Public Analyst stated that the ratio between non-fatty solids and water showed that water had been added. Evidence was given for the defence that it was impossible to fix a standard. Dismissed (*Grocer*, 1910, May 14).

Prosecution under the Merchandise Marks Act for the presence of fluoride. Fine £2 (L.G.B. Report, 1913).

Kensington. Boron trioxide equivalent to 22 grains of boric acid per lb. and injurious to health. It was labelled "Rich preserved cream" and that it contained not more than $\frac{1}{2}$ % of boron preservative. Fine £5. On appeal to Sessions, the conviction was confirmed (*B.F.J.*, 1912, 223; 1913, 88, 106, 128, 142, 161; *Grocer*, 1912, Nov. 9; 1913, May 3, 10).

West London. Boric acid 15 grains per lb. The jar of cream was delivered to the purchaser in a bag, and he was unaware that it was labelled "Preserved cream." Fine 5s. The appeal to the King's Bench, *Batchelour v. Gee*, was dismissed as the vendor had not brought to the mind of the purchaser the presence of a label indicating mixture (*Grocer*, 1913, Oct. 11, 25; *B.F.J.*, 1913, 193, 216; 1914, 87).

Brighouse. Milk nine parts with one part cream. There was a label on the tin: "absolutely pure." Fine £1 (*Grocer*, 1922, April 1).

Kingston-on-Thames. Formaldehyde 0.01 %. Fine £2 for selling it, and £2 for failing to label it (*Grocer*, 1922, July 29; *Analyst*, 1922, 47, 513).

Aberdeen. Adding colouring matter, namely annatto, and selling the coloured cream. Fine £1 in each case. The fat was only 7.7 % (*B.F.J.*, 1923, 85).

Birkenhead. Artificial emulsion of butter. It did not turn sour within a week. The defendant said he had no emulsifier, and used no butter. Dismissed (*B.F.J.*, 1928, 45).

London, Mansion House. Fat not derived from milk 95 %. It was from "Fruit salad with cream" and was little else but whipped margarine. Fine £15 (*B.F.J.*, 1930, 5; *Grocer*, 1930, Dec. 21).

Coventry. Boric acid 0.25 %. Fine £2 (*Grocer*, 1930, Aug. 30).

Birmingham. Boric acid 0.12 %. Fine 10s. (*Grocer*, 1930, Sept. 13). (See also Addenda, p. 577).

PROSECUTIONS FOR PRESERVED CREAM. *London, Marlborough Street.* Boric acid 23.8 grains per lb. and injurious to health. The magistrate dismissed the case, holding that preserved cream was a mixed article and therefore there had been no mixing with an injurious ingredient. On appeal, *Haig v. Aerated Bread Co.*, the High Court directed the magistrates to convict. It was held that a label was no defence in the case of an article injurious to health, and that the only possible defence was that the vendor was ignorant of the addition, and could not with reasonable diligence have obtained the knowledge (*B.F.J.*, 1916, 306).

Kensington. Boric acid 19.7 grains per lb., and injurious to health. Fine £10. The appeal to the High Court, *West London Dairy Co. v. Dawes*, was dismissed (*Grocer*, 1915, Dec. 11; *B.F.J.*, 1916, 251, 308).

PROSECUTION FOR THICK RICH STERILISED CREAM. *Newport, Isle of Wight.* Milk fat 22.34 %, milk solids other than milk fat 8.49 %, water 69.17 %. Rich cream should contain 40–70 % of fat. Evidence was given for the defence that cream containing more than 25–30 % of fat could not be sterilised. The magistrates, though they considered the word “sterilised” should have been in larger print, dismissed the case, owing to the absence of a standard for cream (*Grocer*, 1915, Sept. 11; *B.F.J.*, 1915, 173).

PROSECUTION FOR CREAM THICK AND PURE, STERILISED AND HOMOGENISED. 23–26 % OF BUTTER-FAT. *London, Old St.* Fat only 21.8 %. The tin was sold as it was imported. Costs 3 guineas (*Grocer*, 1928, Sept. 15; *B.F.J.*, 1928, 99).

PROSECUTION FOR THICK CREAM. *Norwich.* Fat 21 % instead of at least 35 %. The tin was labelled “Thick cream, sterilised.” Evidence was given for the defence that there was no standard for cream, that “thick cream” as applied to tinned cream simply meant that it looked thick, and that a cream containing over 25–26 % could not be completely sterilised. Dismissed (*Grocer*, 1929, May 4; *Analyst*, 1929, **54**, 338; *B.F.J.*, 1929, 67).

PROSECUTION FOR CREAM BUNS. *Birkenhead.* Margarine fat 43.3 % of the filling material. The latter also contained 35.0 % of sugar and 21.7 % of water, and was 31 % of the total. No cream was present. The magistrates agreed that what was sold was not prejudicial from the purity point of view, and dismissed the case (*Grocer*, 1928, May 12; *Analyst*, 1928, **53**, 383; *B.F.J.*, 1928, 54).

PROSECUTION FOR BUTTER CREAM SANDWICHES. *London.* Foreign fat, other than butter-fat 100 %. The manufacturers stated the label was intended to mean the flavour of butter cream,

and promised to revise it. Costs £4 (*Grocer*, 1929, March 16; *B.F.J.*, 1929, 46).

PROSECUTION FOR REAL CREAM SHERRY TRIFLE. *North London*. Fat, not milk fat, 29 %. The makers attributed the adulteration to the use of lard to prevent the mixer rusting. Costs 5 guineas (*B.F.J.*, 1930, 47).

PROSECUTION FOR PURE CREAM ECLAIRS. *Aylesbury*. Cream absent; not more than 1 % of the fat in the filling was butter-fat. The counsel for the defence said no one would expect to find real cream in eclairs any more than in peppermint creams, chocolate creams or ice cream, but as the word "pure" was used he must plead "guilty." Fine 10s. (*B.F.J.*, 1930, 77).

CLOTTED CREAM

Clotted Cream. An account of its manufacture, with analyses, was given in the *Journal of the Board of Agriculture* by Sadler in May, 1915 (abstract, *B.F.J.*, 1915, 105, etc.). Richmond has given maxima, minima and average composition of samples examined (*S.P.A.*, 1896, **21**, 89; 1897, **22**, 94; 1898, **23**, 91; 1899, **24**, 200; 1900, **25**, 230). Of the eighteen Birmingham samples analysed, sixteen contained 55–67 % of fat, the two others 53 % and 70 %.

PROSECUTION. *London, Bow Street*. Crystallised boric acid 40.6 grains per lb. Fine £3 (*B.F.J.*, 1916, 454).

ARTIFICIAL CREAM

An Act was passed in 1929 requiring the registration of premises on which artificial cream was manufactured or sold, that the word "artificial" should always precede "cream," and that receptacles containing it should be labelled (*Analyst*, 1929, **54**, 341).

This Act was rendered necessary by the advertisement, about 1927, of emulsifiers for manufacturing "cream" from dried skimmed milk, butter and water. One sample examined in Birmingham revealed its origin by having only 1.3 % of solids-not-fat in the non-fatty part. When it was heated on the water-bath there was an immediate separation of yellow globules, which does not occur with genuine cream. Richardson (*S.P.A.*, 1928, **53**, 334) gave tests for reconstituted cream. If due care is taken in the proportion of the ingredients, its detection is very difficult or impossible.

PROSECUTIONS FOR ARTIFICIAL CREAM. *London, Marlborough Street*. Selling artificial cream as "cream," and conveying it in a receptacle not having "Artificial cream" on it, contrary to the Artificial Cream Act, 1929. It was labelled "Double rich cream, guaranteed pure." Fine £5 in each case. An appeal to Sessions was allowed, as the action was taken, not by a Food and

Drugs Authority, but by a Union not entitled to take proceedings (*Grocer*, 1929, Aug. 17, Sept. 21; *Analyst*, 1929, **54**, 542, 594; *B.F.J.*, 1929, 95, 106).

Wakefield. Selling artificial cream, composed of butter-fats, dried milk and water, as dairy cream. The defendant admitted that the article was artificial cream and said that a gallon of it was made from 5½ lb. best unsalted butter and 3 pints 8 oz. of new milk. He was ordered to pay costs in two cases under the Artificial Cream Regulations, but two cases under the Food and Drugs (Adulteration) Act were dismissed as the sample bottle left with the defendant could be unscrewed without affecting the seal, the red seal not having properly adhered to the metal cap (*B.F.J.*, 1931, 105).

Hastings. Exposing for sale artificial cream in a receptacle not marked "Artificial cream," and using unregistered premises for its sale. Fines £5. (*B.F.J.*, 1932, 16).

PROSECUTIONS FOR SWISS ROLLS (CREAM-FILLED) AND CREAM SANDWICHES. *Swindon*. The prosecutions were under the Artificial Cream Act for the sale of articles containing 30 % and 6 %, respectively, of a cream-like paste containing emulsified fat (a vegetable oil), not derived from cream or milk. The makers claimed that the use of the word "cream" did not indicate dairy cream, which would only keep good a short time. Fine £2 and £8 costs in each case. After several hearings the conviction was quashed on appeal, *Lyons & Co. v. Keating*. The Act said "purporting to be," not "purporting to contain" cream (*B.F.J.*, 1930, 96, 117; 1931, 26, 34, 56; *Grocer*, 1930, Sept. 27; 1931, Feb. 4, March 4, May 9; *Analyst*, 1930, **55**, 689; 1931, **56**, 253, 398).

CHAPTER XIX

FIXED OILS, FATS AND WAXES

Almond oil. Olive oil. Salad oil. Castor oil, castor oil pills. Cod liver oil, cod liver oil emulsion, cod liver oil tablets. Linseed oil. Sweet oil. Nut oil. Lard, lard compound, etc. Dripping, cooking fat. Suet, shredded suet. Beeswax, white wax.

ALMOND OIL

THE Latin name "*Oleum Amygdalæ*" is derived from the old botanical name for the almond, "*Amygdalus communis*." The peach was "*Amygdalus Persica*." They have now been transferred to the genus "*Prunus*." The apricot is "*Prunus Armeniaca*." Apricot and peach kernel oils have been used as substitutes for that from the almond; they are about half the price of it.

In 1914, five out of eighteen Birmingham vendors made this substitution, and in 1925, one out of twelve. In England and Wales during 1905-13, 5.3 % of the samples of almond oil were adulterated.

CONSTANTS OF ALMOND OIL AND SUBSTITUTES

GENUINE SAMPLES.

	Sp. Gr.	Saponification Value.	Iodine Value (Wijs).
Usual range918-.919	192-194	95-100
No. of samples . . .	33	16	24
Extremes917, .920	—	102

SUBSTITUTES.

Usual range918-.920	190-193	97-104
No. of samples . . .	10	5	14
Extremes923	—	95, 124

With one exception, the analytical figures of the substitutes are very similar to those of the genuine oils. The sample with sp. gr. 0.923 and iodine value 124 was probably a mixture of peach kernel oil and poppy seed oil. The acid values of six genuine oils only varied 1.6-2.2. The B.-R. 40° readings varied from 54-57, which is practically the B.P. range.

"Ol. Amyg. Persic.," which is sometimes called "Ol. Amyg. Exot.," is now usually apricot kernel oil, and may be detected by the B.P. acid test. One summons was withdrawn, as the certificate said "peach kernel oil" while the article was chiefly apricot kernel oil. On one occasion a Birmingham inspector noticed that the shop bottle from which he was served was labelled "Ol. Amyg. Persic.,"

and as the vendor labelled the sample "Almond Oil" he was fined for wilfully giving a false label, as well as for adulteration.

The following authors have dealt with almond oil and its substitutes: Bieber (*Y.B.P.*, 1878, 274; 1884, 249), Maben (*P.J.*, 1886, March 20), Allen and Brewis (*B.P. Conf.*, 1900, 359), Lewkowitsch (*S.P.A.*, 1904, 29, 105), Ross and Race (*S.P.A.*, 1911, 36, 263), Pritzker and Jungkunz (*Analyst*, 1928, 53, 102).

PROSECUTIONS. *Birmingham.* Peach kernel oil 75 %. Fine £1 (*B.F.J.*, 1899, 217).

London, Mansion House. Oil other than almond oil 100 %. The bottle was labelled "Oil of Almonds. Ol. amygd. Persic." Dismissed (*B.F.J.*, 1902, 21).

London, Bow Street. Oil other than almond 100 %. After the purchase was completed, the defendant drew the inspector's attention to the label underneath the wrapper:—"Oil of Almonds—Persic." Fine £5 (*P.J.*, 1904, Dec. 24; *B.F.J.*, 1905, 10).

Birmingham. Oil other than almond oil 100 %. Fine £1 for adulteration and £1 for false label (1925 Report).

London, Mansion House. Mineral oil 55 %. Fine £15 (*B.F.J.*, 1919, 15).

Birkenhead. Kernel oil, derived from peach or apricot. The certificate was challenged as it did not state the percentages of foreign ingredients. Defendant ordered to pay £1 10s. (*P.J.*, 1928, May 12; *Analyst*, 1928, 53, 337).

Glasgow. Oil from peach or apricot kernels. For the defence, it was argued that there was no prejudice, because as good an article had been supplied, and that the definition of olive oil must not be limited to the product of the natural almond. Fine £2 (*Grocer*, 1932, Feb. 27; *B.F.J.*, 1932, 26).

OLIVE OIL

Olive oil has been subjected to much adulteration. In 1905 a French newspaper reported on a visit to a railway station near Nice. On the arrival platform there were immense wagons loaded up with nut, cotton, and sesame oils, whilst on the other side there were thousands of neat little wickered carboys labelled, "This oil is our production from the finest olives" (*C. & D.*, 1905, Aug. 19). During 1891-9, 11.6 % of the samples examined in England and Wales were adulterated; during 1901-13, the proportion fell to 3.1 %, and to 0.8 % during 1920-30. Of the 192 Birmingham samples examined three were adulterated with cottonseed oil, and one with arachis oil.

The range of the constants of the Birmingham samples passed as genuine were: sp. gr. (171 samples tested), 79 % of which were 0.916-8, 2 % were 0.914, 5 % 0.915, 13 % 0.919, and 1 % 0.920.

IODINE VALUES OF OLIVE OIL (123 samples)

Iodine value . . .	75	79	80-	82-	84-	86-	88	89	Total.
Percentage of samples.	1	2	8	22	44	20	2	1	100

REFRACTION VALUES OF OLIVE OIL (154 samples)

B.-R. 40° . . .	50.8	51.5-	52.5-	54.0-55.5	65.5	Total
R.I. 40° . . .	1.4599	1.4603-	1.4610-	1.4620-30	1.4636	—
Percentage of samples . . .	1	7	47	44	1	100

The saponification values of fourteen samples were 193-198. Two grades of olive oil have been recommended for the 1932 B.P. with limits of acid values of 2 and 6 respectively, the latter for external preparations.

Analyses of samples of olive oil have been given by Negri and Fabris (*J.C.S.*, 68, ii., 248), Richardson and Jaffé (*J.S.C.I.*, 1905, 534), Thompson and Dunlop (*S.P.A.*, 1906, **31**, 281), Archbutt (*J.S.C.I.*, 1907, 1185).

ANALYSIS. Conno and Rago (*Q.J.P.*, 1929, 332) have recorded that the iodine number falls about three units in a year, but only very slowly afterwards. Some samples of genuine olive oil may give a faint reaction with the B.P. test for sesame oil. The methods for the detection and estimation of arachis oil have been studied by Evers (*S.P.A.*, 1912, **37**, 487). For the detection of olive oil extracted by carbon disulphide the American Olive Oil Committee recommends the silver benzoate test (*Analyst*, 1928, **53**, 497).

About 1920, attention was drawn to the use of tea-seed oil as an olive oil adulterant. Its constants are very similar to those of olive oil, as also are those of "base oil" prepared from a mixture of various oils flavoured with an olive oil of strong aroma. Caulkin (*B.P. Conf.*, 1927, 616) found himself unable to detect tea-seed oil. Bolton and Williams (*S.P.A.*, 1930, **55**, 5) have shown that the iodine value of the unsaponifiable matter of olive oil is higher than that of any other oil, and that this property will detect tea-seed oil.

PROSECUTIONS. *York.* Rape-seed oil. Fine £1 (*F. & S.*, 1894, April 28).

Belvoir. Cottonseed oil 20 %, sesame oil 5 %. The Public Analyst was supported by other analysts, but the Government analysts found no cottonseed oil, but 20 % of sesame oil. The Bench thought that cottonseed oil was present, but inflicted no penalty (*F. & S.*, 1897, April 10).

North London. Cottonseed oil 100 %, sold as a drug. Dismissed on the ground that drugs are not bought at grocers' shops (*F. & S.*, 1897, Sept. 18).

West London. Oil, other than that of olives, 70 %. It was sold

in a flask which was an imitation of an Italian flask. Fine £5 (*B.F.J.*, 1899, 189).

Portsmouth. Sesame oil. Fine £1 (*P.J.*, 1900, Dec.).

North London. Oil extracted from the peanut. Fine £10 (*B.F.J.*, 1902, 203).

Consett. Water 39 %, soap 1 %. Fine £1 (*P.J.*, 1903, Jan. 3 ; *B.F.J.*, 1903, 18).

Chichester. Mineral oil, and no olive oil. In spite of the plea that it was not sold as a food but as a lubricant, the vendor was fined £3 (*P.J.*, 1915, March 27 ; *B.F.J.*, 1915, 78).

Birkenhead. Mineral oil, 7.5 %. The Government analysts found 5.4 %. Fine £1 (*P.J.*, 1916, Sept. 2 ; *B.F.J.*, 1916, 439).

Filey. Arachis oil at least 9 %. The Government analysts reported that the sample was genuine. Dismissed, 11 guineas costs being allowed (*Grocer*, 1922, May 6 ; *B.F.J.*, 1922, 39).

Birkenhead. Soya bean oil. The defendant said he was asked for "oil," not "olive oil," and owing to the doubt the case was dismissed (*Grocer*, 1930, Aug. 23).

SALAD OIL

At one time olive oil in wicker-covered Florence flasks was sold as salad oil, but in recent years the term has been applied to other vegetable oils. In 1919, when olive oil was scarce and dear, there were a number of convictions for the very profitable substitution of paraffin or mineral oil for it. Cases of violent vomiting have been recorded due to the use of mineral oil as salad oil (*B.F.J.*, 1899, 259).

PROSECUTIONS. Cottonseed oil. Fine £3 (*C. & D.*, 1888, Dec. 22).

Kensington. Mineral oil, artificially coloured. Expert evidence was given that salad oils must be vegetable oils, and not mineral oil, which had a different composition and no food value. The objection that the certificate did not state the parts or foreign ingredients was overruled, and the vendor fined £5. In a similar case in which the bottle was labelled "Olivia, a substitute for the highest grade olive oil," the fine was £7 (*Grocer*, 1919, Jan. 18 ; *B.F.J.*, 1919, 6).

CASTOR OIL.

The sale of adulterated oil appears to be uncommon ; none was detected in the fifty-six Birmingham samples examined 1890-1930, nor in the 603 England and Wales samples during 1905-13, while the percentage in 1920-30 was 0.9.

The range of sp. gr. of nineteen Birmingham samples was 0.963-0.967, and two others were 0.961 and 0.962. The acid values of twelve samples were 3 or less, and the saponification values of ten

of them 181-4, extreme figures being 180 and 188. The iodine values of thirteen samples were 84-89.

REFRACTION VALUES OF CASTOR OIL (Fifty samples)

B.-R. 40°	67.1-	68.0-	68.8-	69.5-70	Total
R.I. 40°	1.4704-	1.4710-	1.4715-	1.4720-3	
Percentage of samples	20	50	18	12	100

The rotations of thirty-two samples in a 100 mm. tube varied from 4.2° to 4.6°; two others were 4.0° and 5.1°.

Papers on castor oil have been given by Conroy (*P.J.*, 1889, Nov. 16), Finnemore and Deane (*B.P. Conf.*, 1905, 473), and Stocks (*S.P.A.*, 1923, **48**, 590). Partridge (*S.P.A.*, 1925, **50**, 284) has called attention to the increase in acidity on keeping, and Cocking and Crews (*Q.J.P.*, 1929, 217; *P.J.*, 1929, July 6) have criticised the 1914 B.P. petroleum spirit test.

CASTOR OIL PILLS

No formula is given for such pills in the B.P.; in fact, the term is an absurdity, if not a fraudulent misrepresentation. It is impossible to put more than a trace of castor oil in a pill, and it has been calculated that 400 to 600 pills must be taken to get a medicinal dose, which would be making a meal of them. The name suggests that the oil may be taken in that form without its objectionable taste, while the real ingredient is aloes.

The active ingredient is sometimes phenol phthalein. In 1930 the Public Analyst for Salford interviewed pill-makers and pointed out that "Castor Oil Pills" or "Compound Castor Oil Pills" was a misdescription. They undertook to alter the labels.

PROSECUTION FOR CASTOR OIL PILLS. *Christchurch.* Aloes, rhubarb, peppermint and soap; the certificate stated that injurious results might follow by the purchaser understanding that the active ingredient was castor oil. Evidence was given for the defence that compound rhubarb pills were largely sold as castor oil pills. The vendor was fined 1s. and 19s. 6d. costs, the Bench considering that the evidence of trade custom was only a mitigation of the offence (*Analyst*, 1878, **2**, 105, 146).

Bradford. Croton oil instead of castor oil. Fine 10s. (*F. & S.*, 1894, Aug. 25; *C. & D.*, 1894, Aug. 23).

PROSECUTION FOR COMPOUND CASTOR OIL PILLS. *Lancaster.* About half the pills was aloes; rhubarb, cayenne pepper and soap were detected, but no castor oil. An analyst for the defence, by analysing twenty or thirty pills, had found 1.3 % of castor oil. Fine £2 and £8 costs (*C. & D.*, 1879, April 15; *Analyst*, 1879, **4**, 74).

PROSECUTION FOR COMPOUND CASTOR OIL TABLETS. Castor oil not more than 2.2 %. The tablets were coated with

chocolate and the active ingredient was 7 % of phenol phthalein. Fine £1 (*Analyst*, 1924, **49**, 381).

COD LIVER OIL

There has been little adulteration of this oil detected. During 1903–13, only 0.9 % of the samples examined in England and Wales were condemned, and all but one of the 809 samples during 1920–30 were genuine.

Four of the fifty-four samples analysed in Birmingham were adulterated; the following figures refer to the samples passed as genuine. Of forty-six samples thirty-one had sp. gr. 0.927–8, six were 0.926, and three were 0.929. In thirty-one out of thirty-seven samples the iodine values (Wijs) were 162–171, the extremes being 160 and 180. None of the acid values of twenty-two samples exceeded 2.1, and the saponification values of twenty of them were 185–189.

REFRACTION VALUES OF COD LIVER OIL

B.-R. 40°	. 67.0–	68.0–	68.7–	69.5	70.3–71.0	Total.
R.I. 40°	. 1.4704–	1.4710–	1.5715–	1.4720–	1.4725–30	
Number of samples	. 7	8	10	8	5	38

The polarisation of eight samples in a 100 mm. tube varied from -0.2° to -0.4° , and the Reichert values of six of them were 0.2–0.5.

The above samples gave a purple colour with H_2SO_4 ; the others gave a brown or yellow colour, and were otherwise abnormal—*e.g.*, sp. gr. 0.936, iodine value 123, acid value 9.4, saponification value 198, refraction 61 (1.4665), and Reichert value 1.5.

Papers on cod liver oil and its adulterants have been given by Mann (*P.J.*, 1903, Dec. 5; *Analyst*, 1904, **29**, 93), Liverseege (*S.P.A.*, 1904, **29**, 210; *P.J.*, 1904, July 30), Parry (*C. & D.*, 1905, March 25; *Analyst*, 1905, **30**, 208), Umney and Bennett (*C. & D.*, 1905, Jan. 28, July 4). Williams (*P.J.*, 1912, Dec. 12) has discussed the therapeutic value of the oil, and the nature of its fatty acids.

ANALYSIS. The 1885 B.P. gave a test for liver oils. The addition of a drop of H_2SO_4 to a few drops of the oil on a porcelain slab was to give a violet colour. If the oil was not dry the colour might not appear. The more recent Birmingham samples were tested for vitamin A by adding 1 ml. of arsenic chloride to a drop of the oil in a test-tube and shaking, when a blue colour, changing to purple, was produced (Rosenheim and Drummond, *Biochem. J.*, 1925, 753; *Analyst*, 1926, **51**, 93). See also Wokes and others (*B.P. Conf.*, 1927, 534, 555) and a Report to the League of Nations Health Organisation (*Lancet*, 1928, Jan. 21; *Analyst*, 1928, **53**, 156) showing that the colorimetric (with antimony trichloride) and

biological methods give similar information. Evers has shown that with the antimony trichloride test colours are obtained with old oils, and that the results depend on the method of working (*Q.J.P.* 1929, 556; *Analyst*, 1930, **55**, 287). Dyer and Wokes have found a considerable difference in the amount of colour given by commercial oils (*Q.J.P.*, 1930, 417). The third Report of the Pharmacopœia Commission Sub-committees (1931) should also be consulted.*

PROSECUTIONS. *Thames.* Fish liver oil, other than that of the cod. The Public Analyst stated that his main reason for condemning the sample was that it deposited solid fat on cooling to 0° C. The Government analysts also condemned the sample, but a witness from the National Physical Laboratory considered that, while the sample had not been prepared at quite a low enough temperature, it was a pure oil. It was argued that in regard to a natural product the standards given in the B.P. were not conclusive. The case was dismissed with 3 guineas costs for the prosecution (*C. & D.*, 1904, Jan. 16).

Southwark. Fish oil other than cod liver oil at least 90 %. Three defendants, two herbalists and a fish dealer, were each fined £1 (*P.J.*, 1904, April 30; *B.F.J.*, 1904, 120). The sp. grs. of the samples were 0.9225–0.9263, the iodine values (Hübl) 123–138, B.-R. 25° 72–74.3, the saponification values 184–189, and negative results were obtained with the H_2SO_4 test.

Kensington. Foreign fish oil 20 %. The Public Analyst stated that he had filtered the sample to remove water, that its constants were normal, but that with the B.P. sulphuric acid reaction he obtained a brownish colour. The Government analysts were of opinion that the sample was not pure cod liver oil. After much expert evidence, the magistrate dismissed the case as he considered the evidence showed that the colour test was not altogether reliable, also, that the analyst had not stated if there had been any decomposition (*C. & D.*, 1904, Nov. 19, Dec. 10; 1905, Jan. 21).

Liverpool. Mineral oil (paraffin) 25 %. After warranties had been proved, the importers were fined £20 (*P.J.*, 1910, Jan. 22, Feb. 26, March 26; *B.F.J.*, 1910, 71).

Birmingham. Oil other than cod liver oil 100 %. Fine £5 (1916 Report).

Cod Liver Oil Emulsion. The examination of twenty-six Birmingham samples showed a great range in composition. The variation in sp. gr. was 0.97–1.03; three samples had 20–29 v/v of oil, eight from 30–39 v/v, ten from 40–49 v/v, and five from 50–56 v/v. In the absence of a standard they had to be passed as genuine, except two samples containing 27 v/v and 32 v/v of oil, which were ordered on a prescription as “B.P.C.” According to that formula, 50 v/v of cod liver oil should be present with sucrose

* See Addenda, p. 577.

and gum acacia. Phosphates and hypophosphites were improperly present in seven samples. The usual amount of ash was 0.3–0.4 %, though a sample with mineral addition had 1.5 %. Sucrose was present in about half of the samples tested. There was no definite relation between the sp. gr. and the proportion of oil.

ANALYSIS. The oil may be determined by the Adams or Gerber method, with repeated spinning. Division by 0.92 will convert the weight of the oil into volume, with further correction by the sp. gr. to obtain v/v. The constants of the oil are altered by the extraction.

In the presence of hypophosphites, after the removal of fat by ether from the diluted emulsion, KMnO_4 solution will be decolorised.

PROSECUTIONS FOR B.P.C. EMULSION. *Blackburn.* Cod liver oil 42 % by volume. Fine £2 (*P.J.*, 1921, July 9).

Hull. Cod liver oil 33.4 % by volume. It was ordered by a prescription. The defendant said the article was that usually supplied. Paid costs (*P.J.*, 1927, March 19).

Hull. Cod liver oil 33.5 % instead of 50 %. It was said that the assistant did not notice “B.P.C.” on the prescription. Fine £20 (*B.F.J.*, 1930, 16).

Cod Liver Oil Tablets. In 1928 several samples of these tablets were analysed at Salford. They claimed to be tasteless substitutes for cod liver oil, and to fully retain the important vitamins A and D of the oil. One vendor was prosecuted for a sample of tablets which the Public Analyst (H. H. Bagnall) reported to contain only 3.2 % of oil and oleo-resin, which had the characteristics of an alcoholic extract of cod liver oil. Sugar, gum and other water-soluble matters amounted to 62 %. Vitamin A was absent, and apart from this about 160 tablets would be necessary to obtain a minimum dose of cod liver oil. Biological examination was also made in a pharmacological laboratory, and both vitamins A and D were absent. The vendors were fined £30 and 75 guineas costs (*P.J.*, 1928, April 14; *B.F.J.*, 1928, 44; *Analyst*, 1928, 53, 336).

For another make, it was claimed that each tablet was equivalent to a tablespoonful of cod liver oil, but actually five tablets contained less vitamin A than one *drop* of cod liver oil. Another sample which did contain vitamins claimed to be “equal” to the finest oil, so ignoring the food value of the oil. Another sample, practically free from vitamin A, claimed to be more effective than the oil itself! The vendors of these samples were communicated with and promised to either discontinue the sale or alter the labels.

LINSEED OIL *

The following figures depend on the analyses of fifty-two Birmingham commercial samples of raw linseed oil which were

* See Addenda, p. 577.

passed as genuine, or nearly so. They were not bought under the Adulteration Act.

The sp. gr. of forty-six out of forty-eight samples were 0.931–4. The iodine values (Wijs) were 176–190 in thirty-two out of thirty-five samples, and the B.-R. 40° of thirty-nine out of forty-five samples were 70.2–73.3, corresponding to R.I. 40° 1.4724–44. Two samples were just above the B.P. limit, being 74.7 (1.4752).

ANALYSIS. In addition to the determination of the above values a useful test is to gradually add 100 ml. of boiling water to about 5 gm. of the oil which has been saponified with 50 ml. of E/2 alcoholic NaOH. The resulting liquid should be clear; resin oil or paraffin will give a turbidity. Addition of acid will liberate the fatty acids; the B.-R. 40° figure of the acids of genuine oils is rarely outside the limits 56–58 (1.4633–46).

On heating 2 gm. of the water-free oil in a flat-bottom metal dish on the water bath for two hours there should be no loss in weight, but a gain of 1 % or more (Liverseege and Elsdon, *J.S.C.I.*, 1912, 207; *Analyst*, 1912, **37**, 192). The hexabromide test for linseed oil has been investigated by Hehner and Mitchell (*S.P.A.*, 1898, **23**, 310) and Toms (*S.P.A.*, 1924, **49**, 77).

PROSECUTIONS. *Aylesbury.* Rosin oil 40 %. The fact that the article was in the B.P., and that it was sold by a chemist and druggist, in the opinion of the Bench, showed it was a drug. Fine £2 (*P.J.*, 1900, April, 453).

Horley. Paraffin 10 %. The chemist and druggist who sold it said he had bought it from an ironmonger for his own use, and that it was never used as a drug. Fine £1 (*P.J.*, 1910, March 26).

SWEET OIL

PROSECUTION. *Lambeth.* Mineral oil. It had been sold by an oilman. The inspector said that 70 % of the samples he had bought as “sweet oil” had been nut oil. Three defendants were each fined £2 (*C. & D.*, 1903, Oct. 3). (In Southern England “sweet oil” is often olive oil, while in Northern England and Scotland rape-seed oil is generally understood.)

NUT OIL

PROSECUTION. *Lambeth.* Oil foreign to genuine nut oil 40 %. The inspector said he did not ask for “arachis nut oil.” The magistrate was not satisfied with the certificate and the evidence, and said the 40 % might be a nut oil which the analyst did not recognise. Dismissed, and defendant allowed 2 guineas costs (*C. & D.*, 1910, Dec. 24; *B.F.J.*, 1911, 19).

LARD, LARD COMPOUND

The years 1888 and 1910 were notable for the amount of adulteration of lard with foreign oils and fats. In the former year, no less than 16·8 % of the samples examined in England and Wales were adulterated; in the latter year, the L.G.B. issued a circular on the subject. Divided into periods, the adulteration figures have been, 1886–95, 6·0 %; 1896–1904, 0·6 %; 1905–13, 0·8 %; and 1919–30, 0·2 %. In Birmingham, there have been very few adulterated samples since 1888.

The B.-R. 40° C. were rarely outside the limits 49–51 (R.I. 1·4586–1·4600).

SP. GR. OF LARD (100 samples)

Sp. gr. 99°/15·5°	·8595–	·8600–	·8605–	·8610–	·8615–9	·8626	Total.
No. of samples	3	22	24	43	7	1	100

IODINE (HÜBL) VALUES OF LARD (Fifty-eight samples)

Iodine value	53–	56–	60–	64–	67	Total.
Percentage of samples	5	33	33	27	2	100

MELTING-POINTS OF LARD

Melting-point, ° C.	40–	42–	44–	46–	48–	50–52	Total.
Percentage of samples	2	19	43	21	8	7	100

TITRES OF LARD (Ninety-four samples)

Titre, ° C.	22–	24–	26–	28–9	Total.
Percentage of samples	4	35	48	13	100

It should be noted that there is a difference of about 20° between the melting-point and the titre; some substitutes have a much smaller difference. The Reichert values of forty-three samples were 0·0–4, four others were 0·5–0·8. The B.-R. 40° of the fatty acids obtained from eighteen samples were 33·3–36·6 (1·4476–1·4500).

ACIDITY OF LARD (Forty-seven samples)

Acidity, as % oleic acid	0–	0·2–	0·4–	0·6–	0·8	Total.
Percentage of samples	6	28	30	32	4	100

ANALYSES OF LARDS AND SUBSTITUTES

	Sp. gr. 99/15·5°.	Iodine Value.	B.-R. 40° C.	Melting point. ° C.	Titre ° C.
Lard (leaf)	·8585–8613	46–55	48·3	46–51	30
„ (back)	·8605–8626	56–63	50	44–50	27
„ (commercial)	·8610–8614	56–66	49–52	42–49	24–29
Mutton fat (leaf)	·858	44	49	50	37
Beef fat (leaf)	·858	39	47	44	32
Beef stearine	·857	25	45·3	51	43
Cottonseed oil	·868	108	59	—	—
Lard compound	·865	78–95	53–58	46	20–26

A sample of nut lard had very low figures for refraction (34.7), and Valenta (25), and a high Reichert (8.7).

Only one of the Birmingham samples of lard gave any indication of water, 3.0 % being present ; only one sample out of ten substitutes had water, 10 % being present. The Maximum Prices Order, 1307 of 1918, required that moisture in vegetarian lard should not exceed 0.25 %.

In 1925, some Birmingham traders sold lard compound as "Cooking lard." Enquiries from grocers elicited the information that the term was not generally known in the trade, but was a local one used to describe foreign genuine lard in contradistinction to the superior English lard which is sometimes used for spreading on bread, and described as "Eating lard." "Pastry lard" was stated to be a similar term used to describe somewhat inferior genuine foreign lard. I was also informed that lard compound was chiefly bought by large confectioners, and had little retail sale.

CHANGE ON KEEPING LARD (Four samples)

	Original.	A year later.
Acid value	0.7	5.6
Ester value	195	207
Iodine value (Wijs)	59	50
B.-R. 40°	50.5	51.9
Valenta value	108	75

There have been a number of experiments to determine the effect of feeding on the composition of the fat of the pigs. Willcox and Cranfield used palm kernel meal (*S.P.A.*, 1925, **50**, 323).

Beef and Mutton Fat. Stock (*S.P.A.*, 1894, **19**, 2) suggested a method of determination by weighing the part of lard that was least soluble in ether. A practical difficulty was the large variation found in lard ; one prepared from leaf was 154, and one from the back 58. Muschter and Visser (*Analyst*, 1926, **51**, 353) have described a method and given drawings of the crystals.

ANALYSIS. The sp. gr. was taken in a Sprengel tube in boiling water, the temperature being taken by a short thermometer, and the result corrected to 99° ; 1° C. = 0.0007 sp. gr. Methods for melting-point, titre, and detection of paraffin wax have been given previously (pp. 238, 242).

PROSECUTIONS FOR LARD. *Eddisbury*. Water 18 %. Costs 16s. (*Analyst*, 1882, **7**, 147).

Huddersfield. Beef stearine at least 15 %. It was said that the purpose of the addition was to render the article more firm and capable of crossing the Atlantic. It was labelled "Best leaf lard." Fine £10 (*F. & S.*, 1892, Nov. 5).

Rhyl. Water 11.2 %, membrane 3.8 %. A very dirty sample. Fine 10s. (*F. & S.*, 1897, May 8).

Hastings. Beef fat 25 % ; the Government analysts detected no beef fat. Dismissed with costs (*F. & S.*, 1897, Nov. 20, Dec. 18).

Preston. Paraffin wax 2.3 %. It was stated that the addition enabled a manufacturer to sell an inferior and more fluid lard at the price of the best lard. Fine £2 (*Grocer*, 1908, Dec. 12 ; *B.F.J.*, 1908, 210).

Sunderland. Cottonseed oil 45 %, beef stearine 10 %, water 23 %. Fine £1 (*Grocer*, 1909, Dec. 18).

Stony Stratford. Vegetable fat 25 %, apparently coconut fat. Another analyst, and the Government analysts, reported it to be pure. Defendant allowed 10 guineas costs (*Grocer*, 1910, July 23, Aug. 13 ; *B.F.J.*, 1910, 153).

Margate. Animal membrane 4.2 %. The Government analysts reported 0.25 % of membranous matter, not soluble in ether. Summons withdrawn (*Grocer*, 1910, Aug. 20).

Clerkenwell. Foreign fat 100 %. The wrapper bore the words "Pure vegetable lard," but "vegetable" was in small letters, and "lard" in large letters. Fine £10, as the magistrate did not consider there was sufficient disclosure (*Grocer*, 1910, Oct. 1 ; *B.F.J.*, 1910, 173).

Kensington. Fat of vegetable origin, having the characters of palm oil and coconut oil, 100 %. The defendant had admitted that the article was not lard, but "Palmine." Fine £1 (*Grocer*, 1910, Nov. 19).

Bedford. Fat foreign to genuine lard 25 % (cottonseed oil). The Public Analyst said the iodine absorption (Hübl) was 74.4, that the Halphen and phytosterol tests also gave positive results. Another analyst obtained a Hübl figure of 61.7 and only a slight reaction with the Halphen test. A third analyst confirmed the latter figure, and considered that the reaction was due to cottonseed oil derived from the linoleum cork used for the sample bottle. Dismissed and 10 guineas costs allowed to defendant (*Grocer*, 1912, Oct. 19 ; *B.F.J.*, 1912, 214).

London, Marlborough Street. Fat having the composition of nut lard 92.3 %, water 7 %, salt 0.7 %. Fine £3 (*Grocer*, 1917, June 16).

Harwich. Added water 4.5 %. Dismissed, not sufficient evidence to warrant a conviction (*B.F.J.*, 1923, 36).

Dunmow. Water 4.5 %, being an excess of 4.0 % as compared with well-rendered lard. Ordered to pay costs under First Offenders Act (*B.F.J.*, 1926, 37).

Southend-on-Sea. Ox-blood and vegetable fats. It was said to be sold as a cheaper frying fat. Paid costs (*Grocer*, 1931, Sept. 19).

Chelmsford. Lard compound. Fine £2 10s. (*Grocer*, 1931, Nov. 14).

PROSECUTIONS FOR BEEF LARD. *South Staffordshire.* Cottonseed oil 50 %. The stipendiary thought the name was liable

to deceive the ordinary customer, but as there was no standard he dismissed the case (*B.F.J.*, 1903, 230).

West Hartlepool. Beef or mutton fat 50 %, cottonseed oil 50 %. Evidence was given that beef lard was the recognised designation in the trade of a compound containing about equal parts of beef fat and edible cottonseed oil, and that for some purposes it was more suitable than either lard or dripping. Dismissed by a majority of the Bench (*B.F.J.*, 1917, 85).

PROSECUTION FOR LARDINE. *Guisborough.* Water 25 %. The Public Analyst stated that twenty-eight out of thirty-three samples analysed did not contain water. The magistrates did not consider this was sufficient to establish a standard and dismissed the case. On appeal, *Rudd v. Skelton Co-operative Society* (1911), the case was remitted, and the magistrates were directed to put to themselves the question, "Was the article of the nature, substance and quality demanded?" They must say if it was adulterated, and not decline to fix a standard (*Grocer*, 1910, Oct. 8; 1911, April 1; *B.F.J.*, 1910, 195; 1911, 68).

PROSECUTION FOR LARD COMPOUND. *Swainthorpe.* Water 10.37 %, being 9.37 % excess. Salt 1 %. Fine 1s., as the adulteration had been done by the manufacturer (*B.F.J.*, 1918, 40).

PROSECUTIONS FOR NUT LARD. *London, Old Street.* Applying a false trade description, "Nut Lard," to a mixture of 85 % of cottonseed oil and 15 % of beef stearine. Fine £10 (*Grocer*, 1911, April 1).

Lambeth. Water 20 %. The magistrate said he would make the same standard for nut lard as for butter and margarine, 16 %, and ordered the defendant to pay costs (*Grocer*, 1917, June 23; *B.F.J.*, 1917, 108).

Kensington. Water 18 % excess, boric acid 0.15 %. The Government analysts found 18.2 % of water, and 2 % of boric preservative. Evidence was given that the article was chiefly composed of coconut oil and palm kernel oil, and that 0.5 % of water was sufficient to make it plastic. Fine £2 (*Grocer*, 1917, June 23; *B.F.J.*, 1917, 108).

Essex Quarter Sessions. An appeal against a fine of 10s. for selling as "Sweet nut lard" an article containing 16 % of water was dismissed (*Grocer*, 1917, July 14; *B.F.J.*, 1917, 121).

DRIPPING

The Dripping Order of 1919, No. 511, made under the Defence of the Realm Act, gave the following requirements for dripping.

(a) It shall have been manufactured from raw mutton or beef, fat or bones. (b) It shall not have been manufactured by the acid process. (c) It shall not contain more than 1 % of water and impurities, nor more than 2 % of free fatty acids. These figures

are still used as evidence of what dripping should be, though they are not now legal standards.

Half of the eight Birmingham samples examined in 1903-5 did not comply with the above water standard, the highest amount being 8.8 %. There was a great improvement in 1930, when only one of the thirty-two samples of beef dripping contained water, and that one only 0.1 %.

Of the samples of "dripping and other fats" examined in England and Wales during 1903-13, 6.3 % were adulterated, and 5.6 % of those during 1920-6. In 1907-30, dripping was separately tabulated, and 2.4 % were adulterated.

PROSECUTIONS FOR DRIPPING. *London, Tower Bridge.* Water 5.1 %, when it should have been less than 0.5 %. Costs (*Grocer*, 1912, July 20 ; *B.F.J.*, 1912, 158).

Falkirk. Cottonseed oil 40 %. Quotation was made of the dictionary definition as "melted fat that drips from roasted meat, and which, when cold, is used as butter." Fine £3 (*Grocer*, 1913, Oct. 25 ; *B.F.J.*, 1913, 229).

Bilston. Water 27.4 %, 30.4 %. The retailer was fined £1 for each sample, and the wholesaler was fined £20 (*Grocer*, 1918, April 27 ; *B.F.J.*, 1918, 51).

London, Guildhall. Mineral oil 30 %. The importer was fined £20. (*Grocer*, 1918, Sept. 21).

London, Tower Bridge. Added water 26 %. It was said that though the Dripping Order was dead, it had left its limit of 1 % of water as a legacy which was accepted in all Courts. Fine 21s. (*Grocer*, 1923, March 3).

London, Thames. Free fatty acid 3.1 %, being an excess of 1.1 %. It had been coloured with an organic dye. Evidence was given that a fat which was not edible had been sold as such. Fine £5 (*B.F.J.*, 1925, 115).

Dublin Appeal Court. Rendered beef or mutton fat not containing any of the extractive which falls from roasted meat. The manufacturers gave evidence that their article had been sold as "dripping" for over forty years. The Judge dismissed the case, as he could not hold that what had been sold as dripping for so long a period was not dripping now (*Grocer*, 1927, July 2, 9 ; *Analyst*, 1927, 52, 592 ; see also 1928, 53, 37).

Derry. Fat having the characteristics of cottonseed stearine 25 %. The Government analysts certified the presence of vegetable fat derived from cottonseed. Fine £5 (*Grocer*, 1927, Nov. 19).

Birkenhead. Paraffin wax 8.4 %. The manufacturers, who stated they had bought the stiffening material as "lard wax," were fined £10 for consigning the beef dripping to a stallholder (*Grocer*, 1928, Nov. 17 ; *B.F.J.*, 1928, 115 ; *Analyst*, 1929, 54, 33).

London, Old Street. Free fatty acids 2 %, 4 % and 2.6 %, in

excess of the 2 % allowed. The Public Analyst considered that free fatty acids were injurious as indicating decomposition. The summonses against the two retailers were dismissed, and the manufacturers were fined £5 for the third sample (*Grocer*, 1930, July 12; *B.F.J.*, 1930, 78; *Analyst*, 1930, 55, 572).

PROSECUTION FOR COOKING FAT. *Ballymena*. Cottonseed oil 20.3 %. The defence was that the term "cooking fat" was an invented name, and that the addition of the oil prevented the hardness and brittleness of animal fat. Dismissed (*B.F.J.*, 1904, 42).

SUET, SHREDDED SUET

The Edible Fats (Standard of Quality) Order, No. 658 of 1919 (now obsolete), required that suet should not contain more than 0.5 % of free fatty acids and not more than 0.5 % of water and impurities. Shredded suet was required to contain at least 80 % of fat, not more than 2.5 % of moisture, and no other ingredient except hard grain rice flour.

Of the samples examined in England and Wales, 1908-13, 40 % were adulterated, and 9.3 % of those during 1927-30.

Shredded suet is prepared from suet which had been melted and freed from membrane and other impurities. It is then pressed by hydraulic power through a perforated metal plate, and rice flour blown on to the long strings produced. These are cut into short lengths by rapidly revolving knives; the shreds are then passed over sieves to remove superfluous flour. When packed in cartons, flour may be detached and fall to the bottom.

Six samples of raw suet bought from Birmingham butchers contained 0.8-1.6 % of moisture, and up to 0.5 % of membrane insoluble in ether. Each sample had 98 % or more of fat.

In 1929-30, twenty-one samples of shredded suet were examined; seventeen of them contained 1.0-2.1 % of moisture, the extremes being 0.7 % and 3.5 %. The amount of matter insoluble in ether (starch) in eighteen of them varied 8.9-15.7 %, the extremes being 8.0 % and 21.7 %. The B.-R. 40° of nineteen of them were 46-48 (R.I. 1.4566-80), the other two being 43 and 45. Neither boric acid nor sulphur dioxide was detected.

In each case the articles were sold as mixtures, but several of the wrappers claimed that 1½ lb. of the shredded suet were equal to 2 lb. of raw suet. As the best of them contained about 8 % of fat less than raw suet, these labels were false ones. The attention of the manufacturers was drawn to the misstatements, and they undertook to alter the labels.

PROSECUTION FOR PREPARED SUET. Coconut oil 100 %. It was labelled "Vegetable suet—superior to ordinary suet and more convenient." Fine £2 (*Grocer*, 1911, Jan. 28; *B.F.J.*, 1911, 40).

PROSECUTIONS FOR CHOPPED SUET. *St. Helens.* Added flour 9 %, 14 %, 16 %, 26 %, while 5 % was all that was necessary. The first defendant was fined £1 and the other three £2 (*Grocer*, 1914, June 13).

PROSECUTIONS FOR SHREDDED SUET. *Wolverhampton.* Applying a false trade description, "Guaranteed absolutely pure," to an article containing 19 % and 20 % of ground rice. The cases were withdrawn on the manufacturer undertaking to pay the costs and use a correct label (*Grocer*, 1912, May 18 ; *B.F.J.*, 1912, 119).

Lambeth. Added starch 12.5 %. Dismissed, as the Ministry of Food Order permitted 17.5 % of rice flour (*Grocer*, 1921, Jan. 15 ; *B.F.J.*, 1921, 10).

Lambeth. Ground rice 23 %. It had been served from a box having a label "Shredded beef suet. Refined and coated with rice flour." It was pleaded that as the Order had been revoked there was no standard. The manufacturer gave evidence that 20 % was used at the beginning of the manufacture, but that only about 15 % remained ; and that the flour might be detached and fall to the bottom of the box. Costs 5 guineas (*Grocer*, 1921, Feb. 5 ; *B.F.J.*, 1921, 20, 43).

London, Tower Bridge. Rice starch 25.2 %. Fine £2 (*Grocer*, 1923, Aug. 11 ; *B.F.J.*, 1923, 76).

Stoke-on-Trent. Ground rice 19.2-22.2 % in samples from four retailers. The Government analysts found 15.2 % and 15.5 % in two of the samples. The Public Analyst gave evidence that the average of sixty samples was 15.0 % of added rice flour. Another county analyst said that the average of articles supplied by seven firms was below 15 %, and that 15 % was a very liberal allowance. The stipendiary observed that the defence that the addition was "required" was not valid if an unnecessarily large amount of flour be added. He commented on the large variation in the amount of flour present in different samples, and attributed it to the loose flour in the cartons being unevenly distributed. He fixed the standard as 15 % of desiccated rice flour, with some allowance over that standard, and dismissed the cases (*Grocer*, 1927, Feb. 5, March 5, 26 ; *B.F.J.*, 1927, 29, 39 ; *Analyst*, 1927, 52, 281).

Hull. Rice flour 20 %, being 5 % in excess. The magistrate accepted the standard, and the defendant, who bore a good character, was only ordered to pay costs (*B.F.J.*, 1930, 30).

Lambeth. Ground rice 13.3 %. The defendant said he usually gave a notice of admixture with the article. Fine 10s. 6d. (*Grocer*, 1931, Nov. 14).

YELLOW BEESWAX, WHITE BEESWAX

The use of the terms "yellow wax" and "white wax" in the 1885 (but not in the 1898) B.P., though a literal translation of the

Latin, was unfortunate. It suggested that, in spite of the varied uses of the articles, anything sold under those names should comply with the B.P. descriptions. Also, when "beeswax" was bought from a chemist for medicinal purposes, objection was made that it was not asked for under the B.P. name. In 1892, five of the ten Birmingham samples of "yellow wax" were mixed articles containing notable amounts of paraffin or resin. In 1894 each of four samples of "white beeswax" was largely adulterated, and one of the three samples of "yellow beeswax." In 1897, two of the five samples of "beeswax" were adulterated and one vendor was prosecuted. In England and Wales during 1895-8, 32 % of the samples of "beeswax" were adulterated, and 9 % of the 1907-11 samples.

Hehner (*S.P.A.*, 1883, **8**, 16) has shown that English beeswax consists almost entirely of cerotic acid and myricin (myricyl palmitate). In genuine samples, the ratio between them is as 1.0 to 6.1, and their sum averages, 102.5 %, showing that the assumption that these are the only constituents is not quite correct. The ratio is useful in calculating the composition of adulterated samples.

The 1914 B.P. limits the acid value between 30-40 N. v/w, and the ester value between 124-136 N. v/w for the yellow variety, and allows 30-44 N. v/w for the acid value of white wax; the limits for sp. gr. are 0.958-0.970, and of melting-point 61-64° C. The limits found in seven presumably genuine Birmingham samples were, acid 33-36, and ester 127-133 N. v/w; iodine 7-11 %, sp. gr. 0.964-0.969, and melting-point 62-64° C. Zoneff (*Analyst*, 1927, **52**, 598) has given the ranges of constants of 136 samples of Bulgarian beeswax, and Ikuta those of Japanese beeswax (*S.P.A.*, 1931, **56**, 430). Rottger (*Analyst*, 1890, **15**, 152) found little alteration in composition by bleaching.

ANALYSES OF ADULTERATED BEESWAX AND ROSIN.

	Yellow Beeswax.			White Beeswax.			Rosin.
	I.	IIa.	IIb.	I.	II.	III.	
Acid value, N. v/w.	84	101	22	2	68	22	265
Ester value, N. v/w.	4	8	94	47	20	349	49
Ratio, 1.0 acid to	0.05	0.08	4.3	23	0.3	16	0.18
Iodine value, %	51.0	53.3	9.9	4.9	3.4	13.4	148
Sp. gr. (about 15° C.)	0.960	0.962	0.953	0.932	0.938	0.989	1.079
Melting-point, ° C.	63	58	71	55	54	52	—

CALCULATED CONSTITUENTS.

Beeswax, %	0	6	64	6	16	0	—
Rosin, %	32	37	—	—	—	—	—
Stearic acid, %	—	—	—	—	17	—	—
Tallow, %	—	—	4	11	—	—	—
Japan wax, %	—	—	—	—	—	100	—
Paraffin wax, % (by difference)	68	57	32	83	67	0	—

In genuine samples the relation between acid value and ester value, should be about 1.0 to 3.7 ; in the above samples it varied from 0.05 to 23. Although the first sample was free, or practically free, from beeswax, the sp. gr. and melting-point came within the B.P. limits. IIa and IIb were two parts of a prosecution sample, and were very different in composition ; it illustrates the importance of careful sampling by officers. White sample III was apparently pure Japan wax. The percentages of beeswax given above are maximum values, as *all* the acid, or *all* the ester, whichever gave the smaller value, was calculated to beeswax, while part may have been due to the adulterant.

ANALYSIS. The iodine value is determined on 0.5 gm., or less if much rosin be present. Heat may be necessary to dissolve the wax in the carbon tetrachloride.

The sp. gr. may be conveniently determined by the Westphal balance (Liverseege, *Chemical News*, 1894, Aug. 31), the plummet being exchanged for (1) the 1.0 weight, (2) a light scale pan with a hook underneath, to which is attached, (3) a spindle-shape brass sinker, weighing about 7 gm., immersed in a beaker of water, and (4) a piece of lead on the scale pan to produce equilibrium. A cube of wax weighing less than 6.5 gm. is smoothed and polished to prevent air bubbles sticking to it. The wax is first put on the scale pan, and the sp. gr. weights adjusted ; the sinker is then stuck in the wax, put in the water and the weights again adjusted. In the balance used, 1.0 sp. gr. weight weighed 6.48 gm., but there is no need to calculate weights to gm.

$$\text{Sp. gr.} = \frac{1 - \text{first reading}}{\text{second reading} - \text{first reading}}.$$

Rosin, or other substance heavier than water, can be done in the same way. It may be necessary to warm the sinker before sticking into the substance, but the temperature of observation must be about 60° F. The presence of air spaces in the wax is provided against by cutting the cube from wax that has been melted and allowed to cool.

Rosin should be tested for by heating a small piece of the wax with strong HNO_3 in a test tube, diluting with an equal volume of water, and adding excess of ammonia. With pure wax a yellow colour will be given, but a small proportion of rosin will give a red colour.

The presence of any notable amount of paraffin is obvious during saponification, but its determination is unsatisfactory. Heat 5 gm. with about 100 ml. of commercial H_2SO_4 , cool, pour away acid, and repeat once or twice. Boil the cake of wax in a beaker with water made alkaline with KHO. Cool, put cake in beaker with petroleum ether, and heat till dissolved, filter out carbon, and evaporate

filtrate and dry in weighed dish. Wilkie (*S.P.A.*, 1917, **42**, 200) has given a method for determining unsaponifiable matter in waxes.

CALCULATION. 1 ml. of normal KHO = 0.3965 gm. cerotic acid, or 0.6769 myricin. Let B = % beeswax, I = iodine value, C = acid value N. v/w, and M = ester value, N. v/w.

$B = 2.8 C$, or $B = 0.788 M$, whichever is smaller.

Resin, % = $0.377 (C - 0.27 M)$, or $0.68 (I - 0.1 B)$.

Stearic acid, % = $0.284 (C - 0.27 M)$.

Tallow, % = $0.28 (M - 3.7 C)$.

If resin and paraffin only are present :—

Resin = (sp. gr. — .909) 590.

PROSECUTIONS FOR BEESWAX. *Bristol.* Solid paraffin with a little fatty matter, coloured with a coal tar dye. Fine £1 (*F. & S.*, 1894, Nov. 24).

Newport. Paraffin 87 %, resin 8 %, and only 5 % beeswax. For the defence it was contended that beeswax was used for cleaning floors and furniture, and could not be described as a drug as it was only used in pills for binding purposes. Paid costs (*F. & S.*, 1895, Sept. 7 ; *P.J.*, 1895, Sept. 7).

Bingley. Added fatty matters, chiefly Japan wax and stearic acid, 75 %. Fine 5s. (*F. & S.*, 1895, Nov. 2).

Ashford. Paraffin 50 %. Evidence was given that yellow wax was used directly in the preparation of twelve B.P. articles, and indirectly in eight others. Fine 10s. (*F. & S.*, 1896, Feb. 1).

Cranbrook. Foreign matter—to wit, paraffin 50 %. This sale by a grocer was dismissed, and led to the appeal case, *Fowle v. Fowle* (1896, p. 48).

Birmingham. Paraffin wax 25 %, resin 10 %. As it was the first case of the kind, and as the chemist did not dispense medicine, a fine of only 1s. was imposed (*F. & S.*, 1897, May 29).

Deal. Paraffin wax 30 %. The article had been sold by a chemist, but the magistrates dismissed the case as it had not been proved to their satisfaction that beeswax was a drug (*B.F.J.*, 1902, 44).

Lambeth. Rosin 40 %, paraffin wax 15 %, and only 45 % of beeswax. Fine £1 (*B.F.J.*, 1907, 117 ; *P.J.*, 1907, June 22).

Lambeth. Paraffin wax 20 %, resin 5 %. The purchaser bought four tablets ; two were taken to the Public Analyst, one was kept and the other given to the vendor. It was stated that the tablets were parts of two separate consignments. The summons was dismissed, the magistrate remarking that each tablet should have been divided into three parts (*P.J.*, 1907, July 20).

London, Thames. Paraffin wax 66 %. It was bought at a drug stores, and the vendor was fined 10s. and costs. Another vendor whose sample had 90 % of paraffin wax was fined £1 (*P.J.*, 1913, Jan. 11).

PROSECUTIONS FOR WHITE WAX. Paraffin wax 75 %, 90 % and 95 %, respectively, not being according to the B.P. The samples were bought from chemists, and each was ordered to pay £1 (*F. & S.*, 1894, Nov. 3).

Skipton. Paraffin wax 70 %. Case dismissed as the B.P. quality had not been specified (*C. & D.*, 1895, Feb. 23).

Pontefract. Paraffin 70 %. The defendant stated that there were two kinds of white wax, viz., that recognised by the B.P., which was not absolutely white, and a second kind used for other than medicinal purposes, and that it was the latter, which was not a drug, which he sold. Case dismissed (*F. & S.*, 1895, March 2 ; *C. & D.*, March 2).

CHAPTER XX

NITROGENOUS FOODS

Meat products :—Minced meat, mincemeat, steak, lamb, tongue, kidneys, tripe, brawn, potted meat, etc., pork pie, chicken and ham roll, pork and beans, bacon, caviare, sausage, beef sausage, German sausage, sausage rolls, black pudding, polony. Meat extracts. Fish products :—Sardines, prawns, shrimps, lobster, fish pastes. Eggs, liquid egg flour. Custards, custard powders. Cheese :—Margarine cheese, Cheddar, Cheshire, Dutch, Gorgonzola, milk cheese, cream cheese. Cheese sandwiches.

MEAT PRODUCTS

In 1908, reports by Buchanan and MacFadden to the L.G.B. (Nos. 3, 4 and 6 ; *B.F.J.*, 1908, 23 ; *Analyst*, 1908, **33**, 382) called attention to the presence of boric acid in imported tripe, tongues, kidneys, hams, bacon, and also in canned or glass-packed potted meat, etc. In the following year a report (No. 9, *Analyst*, 1909, **34**, 470) was made by Buchanan and Schryver on the use of formaldehyde for meat, giving a method of determination. Callow (*S.P.A.*, 1927, **52**, 391) showed that smoking of bacon, etc., might introduce formaldehyde.

In 1896 a number of samples of meat foods bought in Birmingham contained boric acid. In 1905 the examination of three kegs of imported tripe indicated the presence of 30–105 grains of boric acid per lb. and in 1907 an imported pig's kidney, after soaking in water for five minutes, contained 190 grains ! Four samples of polony, in 1922, had 18–24 grains of boric acid per lb. ; other figures for them were : water 36·6–59·5 %, protein 12·1–16·1 %, fat 15·8–35·3 %, and calculated meat 64·3–99·5 %.

The official reports for England and Wales give figures which show that the percentage of adulteration was as follows. The Preservative Regulations came into force in 1927 :—

ADULTERATION OF MEAT FOODS IN ENGLAND AND WALES

	1906–13.	1920–6.	1927–30.
Potted meat	19·3 %	10·3 %	6·0 %
Meats, not canned nor potted.	25·0 %	8·1 %	5·2 %
Meat pies	23·2 %	—	—
Meat pies and rolls	—	19·0 %	1·8 %
Sausages	29·0 %	—	—
Sausages and polonies, etc.	—	17·0 %	10·1 %
Mincemeat	12·4 %	8·6 %	1·7 %

ANALYSIS. The same methods may be used as are given below under "Sausage." Manley and Sutton (*S.P.A.*, 1930, 55, 11) have given methods and analyses of potted meats; they suggested that reasonable standards would prohibit the addition of starch and the presence of more than 70 % of water. Meat pastes, which are intended to be spread on bread and butter, require the presence of a starchy ingredient.

PROSECUTIONS FOR MINCED MEAT. (The Scottish Preservative Regulations permit the presence of 450 parts of sulphur dioxide per million in minced butcher's meat during June, July, August and September.)

Derry. Sulphur dioxide 10 grains per lb. Evidence was given that it was likely to cause dyspepsia, and its presence would mask signs of decay in the meat. Fine £10 (*B.F.J.*, 1925, 116).

Aberdeen. Boron preservative equal to 800 parts of boric acid per million. It also contained more than 450 parts per million of sulphur dioxide. Vendor fined (*Grocer*, 1927, Sept. 3).

Greenwich. Sulphur dioxide 1,500 parts per million. The defendant said that he had used the preservative for years to give the meat a good colour. Fine £2 (*Grocer*, 1927, Dec. 24).

PROSECUTIONS FOR MINCEMEAT. *Stockport.* Salicylic acid 4.3 grains per lb. The retailer proved a warranty, and the wholesaler was fined £2 for giving a false warranty. He appealed in vain to Quarter Sessions (*Grocer*, 1923, April 14; *B.F.J.*, 1923, 29, 33).

Coleraine. Excess of sulphur dioxide. Fine 30s. (*Grocer*, 1930, Aug. 9).

PROSECUTIONS FOR STEAK. *Brentford.* Boric acid 14 grains per lb. The assistant admitted dusting the meat with a powder. Fine £2 (*B.F.J.*, 1927, 96).

Kingston. Sulphur dioxide 100 parts per million. It was said to be due to "Fakers," a brown paper compound burnt to clean and dry the ice safe. Paid 5 guineas costs (*B.F.J.*, 1929, 8).

PROSECUTION FOR LAMB. *Croydon.* Sulphur dioxide 0.025 %. The assistant said the meat had been "just dusted over a little" and was to be made into sausages. He was fined £1 and his employers £5 (*B.F.J.*, 1929, 37).

PROSECUTION FOR NEW ZEALAND LAMB. *Plymouth.* Sale of South American lamb, to the prejudice of the purchaser. Evidence was given that at the date of sale there was no New Zealand lamb on the market. Fine £5 (*B.F.J.*, 1929, 37).

PROSECUTIONS FOR TONGUE. *East Dereham.* Boric acid 67 grains per lb. Pickled tongues had been packed in a glass jar. Fine £10 (*B.F.J.*, 1906, 227).

Wood Green. Boric acid 90 grains per lb. The tongue had been supplied to the vendor ready cooked in an open tin. Paid costs (*B.F.J.*, 1908, 35).

PROSECUTION FOR KIDNEYS. *London, Old Street.* Boric acid 63 grains per lb. The kidneys were stated to have been sold as imported. Fine £2 (*B.F.J.*, 1908, 70, 74).

PROSECUTIONS FOR TRIPE. *Stoke.* Boric acid 0.2 %. A system of washing tripe in a weak solution of boric acid had been adopted to make it look white and more appetising; it was afterwards washed with water. Fine £2 (*B.F.J.*, 1928, 80).

East Ham. Sulphur dioxide 180 parts per million. It was said to be "due to a tank of preservative water." Fine £5 (*B.F.J.*, 1930, 17).

PROSECUTIONS FOR BRAWN. *Carnarvon.* Boric acid 31 grains per lb., and a large number of bristles and micro-organisms. Ordered to pay costs (*B.F.J.*, 1910, 133).

Brentford. Boric acid 0.398 %, artificially coloured with a pink coal tar dye. Fine £2 (*Grocer*, 1906, July 21).

PROSECUTIONS FOR POTTED MEAT. *Skipton.* Boric acid 154 grains per lb. Fine £5 and costs (*Grocer*, 1914, Aug. 29; *B.F.J.*, 1914, 178).

Nottingham. Starchy filler 39.12 %. The meat contained 75 % of water. The inspector stated that a large number of samples of potted meat had contained 78-94 % of meat and no starchy filler. The defendant stated that shortly after the sample was taken the maker changed the name to "Meat paste." Summons dismissed under the Probation Act (*Grocer*, 1926, March 20; *B.F.J.*, 1926, 40).

Nottingham. Water in excess 36.15 %. Fine £5 (*B.F.J.*, 1930, 28).

Mansfield. Starch 14.8 %. Fine 21s. (*Grocer*, 1931, May 2; *B.F.J.*, 1931, 60).

PROSECUTIONS FOR POTTED HAM. *Brentford.* Boric acid 52 grains per lb., and artificially coloured with oxide of iron. Penny tins had been bought. Fine £5 and 50 guineas costs (*Grocer*, 1906, July 21; *B.F.J.*, 1906, 159).

Docking. Boric acid 2.4 % and injurious to health. Fine £5 (*Grocer*, 1907, Aug. 3; *B.F.J.*, 1907, 143).

PROSECUTION FOR POTTED CHICKEN, HAM AND TONGUE. *Kensington.* Boric acid 25 grains per lb., including the layer of fat, or 34 grains, excluding it. It was suggested that the layer of fat was not edible, and therefore should not be taken into account. Fine £5 (*B.F.J.*, 1913, 37).

PROSECUTION FOR HAM AND TONGUE PASTE, AND HAM AND BEEF RISsoles. *West London.* Boric acid 44 grains and 21 grains per lb., respectively. The vendor was fined £2 and 10s. (*Grocer*, 1913, April 26; *B.F.J.*, 1913, 99).

PROSECUTION FOR POTTED TONGUE PASTE. *Newcastle.* Bone phosphate 10.5 %. The Bench was satisfied that the addition was with the intention of fraudulently increasing the bulk and fined

the manufacturers £20. About 4,000 tins were withdrawn from sale (*Grocer*, 1931, Aug. 8; *B.F.J.*, 1931, 79).

PROSECUTION FOR HAM AND CHICKEN PASTE. *Nottingham.* Starchy filler and water 41 %. The defendant undertook only to use meat, and was fined 10s. (*Grocer*, 1921, June 25).

PROSECUTION FOR BEEF PASTE. *St. Helens.* The defendant was fined £1 for refusal to sell. He had been twice convicted previously (*B.F.J.*, 1932, 14).

PROSECUTION FOR PORK PIE. *Amptill.* Boric acid 28 grains per lb. It was said to have been insufficiently mixed. Fine £10 (*Grocer*, 1924, Jan. 26).

PROSECUTION FOR STEAK AND KIDNEY PIES. *Norwich.* No kidney was present. The pies were exposed for sale with a printed label. Evidence was given that a mistake had been made in labelling the pies, and the case was dismissed (*B.F.J.*, 1931, 110).

PROSECUTION FOR CHICKEN AND HAM ROLL. *Salford.* Beef protein 8.5 %, beef fat 13.3 %, starch and crude cellulose 13.3 %, mineral matter (including 0.3 % boron preservative) 2.2 %, and water 62.7 %. The meat contained in it was beef 42 %; starchy filler 26 % and excess water 32 %. The offence was admitted, and it was pleaded that no one was deceived. Fine £10 (*Grocer*, 1926, Sept. 4; *Analyst*, 1926, 51, 515; *B.F.J.*, 1926, 93).

PROSECUTION FOR CHICKEN ROLL. *Salford.* Pork protein 16.1 %, pork fat 15.8 %, starch and crude cellulose 11.7 %, mineral matter 3.1 %, and water 53.3 %, corresponding to pork 75 % and starch filler 25 %. The vendor, who sold it as he received it, was fined 5s. The manufacturers, who admitted that no chicken was present, were prosecuted for aiding and abetting, but the summons was dismissed as they had not sold directly to vendor. They altered the label to "Delicious Roll. . . Chicken and ham flavoured" (*Grocer*, 1926, Sept. 4; *Analyst*, 1926, 51, 516; *B.F.J.*, 1926, 94).

PROSECUTION FOR PORK AND BEANS. *London, Old Street.* Meat not more than 0.9 %. The tin was labelled "pork" and "BEANS" in the same size lettering, and the Public Analyst expected quite 15 % of meat. The defendant proved a warranty and the case was dismissed (*Grocer*, 1928, March 12; *B.F.J.*, 1928, 59).

PROSECUTION FOR BACON. Boric acid 7.7 grains per lb. Fine £15. The case against a vendor who had used some of the bacon for making sausages was dismissed on payment of costs (*Grocer*, 1929, Oct. 19; *B.F.J.*, 1929, 113).

CAVIARE *

Analyses of caviare have been given by Hinard (*Analyst*, 1923, 48, 552).

PROSECUTION. *Bow Street.* Boric acid 0.28 % and 0.25 %.

* See Addenda, p. 577.

respectively. It was said that the articles had been sold as imported, and that without boric acid it would not keep. Each vendor paid costs (*Grocer*, 1929, Nov. 9; *B.F.J.*, 1929, 125; *Analyst*, 1930, 55, 40).

SAUSAGE

The composition of seventeen samples of Birmingham pork sausage, analysed 1922-4, was as follows:—Water 41.0-53.7 %, ten of them being between 45 % and 48 %; fat 21.4-31.9 %, protein (nitrogen $\times 6.25$) 5.7-10.6 %, thirteen of them being between 7.5 % and 8.9 %. On the assumption that four times the protein added to the fat gives the amount of meat present, nine of the samples contained 55-60 %, and the other eight, 62-74 % of meat.

The above assumption was tested by the analysis of a sample of sausage which was stated by the makers to contain 46.1 % lean pork, 23.1 % of fat pork and 28.8 % of cooked rice, the remainder being salt, spice and preservative. The sample was found to contain 8.6 % of protein and 35.4 % of fat; on the stated assumption, 69.8 % of meat was present, a result very near that given by the makers.

In 1918 a war-time Retail Price Order (No. 284) was made, fixing the price of sausage containing not less than 50 % of meat. In the next year a vendor was prosecuted at Marylebone under this Order. The Public Analyst reported that it contained only 30 % of meat, of which 13 % was fat. Another analyst found 50.17 % of meat, and the Government analysts 44 %. Details of the analytical figures of the two later analyses were given, mean results being protein 9.7 % and fat 12.4 %. From these figures the above-given formula indicates 51.2 % of meat, while the manufacturer stated that 52 % of meat had been used for the sausage. The magistrate considered that the amount of meat present was under 50 %, and fined the vendor £75, with 25 guineas costs (*Grocer*, 1919, June 28; *B.F.J.*, 1919, 69).

A single sample of Birmingham "Fish sausage" contained 75.2 % of water, 13.4 % of protein, 0.3 % of fat, and 3.1 % of ash.

Sausage was first tested for boric acid in Birmingham in 1908, when 44 % of the samples were free from that preservative and 16 % contained 35 grains per lb. or more. Little action was taken in the matter, and during 1922-6 only 14 % of the samples were free from boric acid, and 22 % contained 35 grains per lb. or more. The Preservative Regulations came into force in 1927, and all the samples examined 1927-9 were free from boric acid.

The Preservative Regulations permit the use of sulphur dioxide in sausages up to 450 parts per million, if the article is labelled or a notice is exhibited in the shop in a conspicuous place. In practice, this alternative has proved unsatisfactory, as samples bought as

"Preserved sausage" from shops displaying the notice have proved to be free from preservative, and therefore not of the nature of the article demanded. The maker has sometimes supplied the shopkeeper with preserved sausages, and at others with an article free from preservative, while the vendor has assumed that preservative was always present. If the makers were required to label all preserved sausage, mistakes of this kind should not occur; the articles are not identical, and the distinction should be preserved.

The Birmingham results for 1928-31 illustrate this. Only seventeen of the thirty-two samples bought as "Preserved sausages" contained appreciable amounts of sulphur dioxide, while twenty-one of the forty-seven samples of "Sausages" contained this preservative. Only two of the twenty-eight samples exceeded the limit, 500 and 840 parts per million being present respectively. Fifteen samples had 240-430, twenty-one had 90-210, and in three samples traces only were detected: duplicate samples are not counted. A sample of sausage, containing 290 parts of sulphur dioxide per million, after frying for fifteen minutes contained 240 parts.

A sample of sausage, which contained 400 parts per million of sulphur dioxide, on re-examination when it had become mouldy after twenty-six days indicated 460 parts. This agrees with the experience of Black and Warren (*S.P.A.*, 1928, **53**, 132), that putrefaction in meat may increase the apparent SO_2 content by 75-100 parts per million.

H. O. Jones (*S.P.A.*, 1928, **53**, 138) stated that on the manufacturing scale it is difficult to obtain an absolutely uniform mixture of sausage and sulphite, and that when 450 parts of sulphur dioxide are added there is an almost immediate loss of about 150 parts per million. Drake-Law has reported (*S.P.A.*, 1927, **52**, 353) a similar loss with cooked meats. Hinks (*S.P.A.*, 1928, **53**, 128) stated that the sulphur dioxide in sausages halved itself in two days. According to Campbell (*Analyst*, 1924, **49**, 532), raw meat kept in hermetically sealed bottles below 50° F. showed no serious loss of sulphur dioxide in periods up to six weeks.

ANALYSIS. Weigh 5 gm. in a metal dish, and determine *moisture* lost on drying. Grind up residue with petroleum ether, and thoroughly wash dish, decanting through a folded filter placed in a Soxhlet extractor; extract two hours and weigh *fat*. Determine *nitrogen* in the residue by Kjeldahl method. Determine *starch* by treating 20 gm. with 100 ml. 1.5E alcoholic KHO in a 250-ml. conical beaker. Filter through Gooch crucible and wash with warm alcohol till filtrate is colourless. Transfer entire contents of crucible with hot water to original flask, neutralise, dilute to 200 ml., add 15 ml. 10E.HCl, and place in boiling water bath for three hours. Reducing sugar, determined by Fehling solution, $\times 0.9 = \text{starch}$.

Methods for the analysis of sausages have been given by Stubbs and More (*S.P.A.*, 1919, **44**, 125) and Stokes (*S.P.A.*, 1919, **44**, 127); the latter gives analyses of samples.

PROSECUTIONS. *Trowbridge.* Boric acid 105 grains per lb. Fine £1 (*B.F.J.*, 1905, 106).

London, Marlborough Street. Boric acid 22 grains per lb. Fine £2 and £5 5s. costs. The defendant appealed to Quarter Sessions, and his appeal was dismissed with costs (*Grocer*, 1908, Dec. 5, 12, 19; 1909, May 22, June 12; *B.F.J.*, 1908, 205; 1909, 105). For a similar appeal see *Grocer*, 1918, Feb. 16, April 13; *B.F.J.*, 1918, 24, 55).

Eastbourne. Water 47.6 %, fat 12.7 %, dry starch 14.7 %, and dry fat-free meat 25 %. The Public Analyst considered the sample was 66 % of meat and 34 % of bread dressing. The case was dismissed, as there was no standard for sausages (*Grocer*, 1915, July 31; *B.F.J.*, 1915, 176).

Eastbourne. Water 53 %, fat 10.3 %, dry starch 27.4 %, dry meat 9.0 %, the original composition being probably 20 % of meat and 80 % of bread. Fine £1 (*B.F.J.*, 1915, 178).

Woburn. Boric acid 28 grains per lb. The Government analysts found 41 grains, but remarked that the sample had lost water by evaporation. The label—"These sausages contain a small percentage of boron preservative"—was described as not being truthful. Fine £20 (*Grocer*, 1924, July 26; *B.F.J.*, 1924, 89).

Birkenhead. Meat 43 %, starch filler 31 % and 26 % added water, being deficient in meat to the extent of at least 7 %. Another analyst found 52 % of meat. The foreman stated he used 53 % of meat, and the clerk said during the week each 54 lb. of meat represented 100 lb. sausage. Case dismissed (*B.F.J.*, 1925, 49).

Derry. Sulphurous acid 0.082 %, equivalent to 5.7 grains per lb. Fine £10 (*Grocer*, 1926, June 9; *B.F.J.*, 1926, 80).

Brentford. Boric acid 1,970 parts, and sulphur dioxide 27 parts per million. Fine £20 (*Grocer*, 1927, Dec. 24).

Ealing. Selling sausages containing sulphur dioxide preservative without the sausage being labelled to that effect as required by the Preservatives in Food Regulations. Two vendors were each fined £5 (*B.F.J.*, 1928, 19).

Wigan. Boric acid 14 grains per lb., contrary to the Preservative Regulations. Fine £5 (*Grocer*, 1928, July 21).

Newmarket. Benzoic acid 115 per million. Fine £2, and also £2 for exposing them for sale unlabelled (*B.F.J.*, 1929, 96).

Halifax. Sulphur dioxide 599 parts per million. Fine £1 (*Grocer*, 1930, Aug. 24; *B.F.J.*, 1930, 89).

Kensington. Boric acid 7.931 grains per lb. Fine £10 (*Grocer*, 1931, June 20).

PROSECUTIONS FOR BEEF SAUSAGES. *London, Marylebone.* Boric acid 116 grains per lb. It was said that a mistake had been made in the quantity of preservative added, and that it had not been mixed properly. Fine £1 (*B.F.J.*, 1925, 90).

Aberdeen. Sulphur dioxide 461 parts per million, being in excess of the 450 parts allowed. Fine 25s. (*Grocer*, 1927, Sept. 3).

London, Marylebone. Selling sausage containing sulphur dioxide preservative without declaration of its presence, to the prejudice of the purchaser. Summons dismissed, being taken under the wrong section (*B.F.J.*, 1928, 110; *Analyst*, 1928, **53**, 649).

Gosport. Boric acid 0.08%. The vendor suggested that the boric acid must have been in the seasoning or in the skins, but neither contained it. It was afterwards admitted that boric acid had been sprinkled on the sausage meat. Fine £2 (*Grocer*, 1932, Jan. 9; *B.F.J.*, 1932, 10).

PROSECUTIONS FOR GERMAN SAUSAGE. *Westminster.* Boric acid 21 grains per lb. Ordered to pay 12s. 6d. costs (*B.F.J.*, 1915, 119).

PROSECUTION FOR SAUSAGE ROLLS AND SAUSAGES. *London, Bow Street.* Boric acid 28 and 43 grains per lb. respectively. The fines and costs amounted to £11 15s. (*Grocer*, 1914, Jan. 10; *B.F.J.*, 1914, 38).

PROSECUTION FOR BLACK PUDDING. *Castle Eden.* Boric acid 0.6 %. The vendor was ordered to pay costs only, on promising to give £5 to a hospital (*B.F.J.*, 1927, 113).

PROSECUTION FOR POLONY. *Leicester.* Boron equivalent to 0.017 % of boric acid. The preservative was attributed to the washing of the skins in water containing it. Fine £1 (*Grocer*, 1931, Sept. 5).

MEAT EXTRACTS

Methods for the separation of the different nitrogenous bodies contained in meat extracts have been given by Rideal and Stewart (*S.P.A.*, 1897, **22**, 228), Allen and Searle (*S.P.A.*, 1897, **22**, 258), Emery and Henley (*Analyst*, 1919, **44**, 292), and Berk and Schneider (*Analyst*, 1923, **48**, 551); the latter paper and also one by Cook (*Analyst*, 1914, **39**, 444) give the distribution of nitrogen in various extracts. Reference should also be made to the Report of the joint meeting of the Society of Public Analysts and the Biochemical Society on the subject (*Analyst*, 1915, **40**, 310). The particular determination of the amounts of creatin and creatinin is of importance, as they are absent from yeast extracts. Barschall (*Analyst*, 1907, **32**, 48), Hehner (*Analyst*, 1907, **32**, 292), and Chapman (*S.P.A.*, 1909, **34**, 475) have examined the Jaffé method, and Grindley and Woods (*Analyst*, 1907, **32**, 171), Emmett and Grindley (*Analyst*, 1908, **33**, 51), and Cook (*Analyst*, 1909, **34**, 395) that of

Folin. Detailed analyses of meat and yeast extracts have been given by Graff (*Analyst*, 1904, **29**, 194), Cook (*Analyst*, 1911, **36**, 104), and Emery and Henley (*opus cit.*). Elsdon (*S.P.A.*, 1924, **49**, 213) gives the composition of meat extract according to various authorities.

Buchanan and Schryver have reported on meat extracts which had been kept in tins from four to seven years; the methods of analysis used are given. One badly soldered tin had over 21 grains of tin per lb., the other five had under 2 grains per lb. (L.G.B. Report, No. 1, 1906).

Of the samples of meat extracts examined in England and Wales 1906-12, 11.0 % were reported adulterated, but only two of the 211 samples examined 1920-30.

PROSECUTION FOR MEAT EXTRACT. *Liverpool.* Yeast extract about half its weight. Fine £5 (*B.F.J.*, 1904, 44).

PROSECUTION FOR BEEF AND VEGETABLE CONSOMMÉ. *Lewes.* Mineral matter 60 %, about two-thirds of which was salt. Case dismissed, each party to pay their own costs (*Grocer*, 1910, June 11; *B.F.J.*, 1910, 107).

FISH PRODUCTS

Essery has given drawings and descriptions of fish-scales as a means of identifying the fish used in manufactured products (*S.P.A.*, 1922, **47**, 163). Büttner and Miermeister have given a test to detect artificial crab colour (*Analyst*, 1929, **54**, 546). Cox (*S.P.A.*, 1925, **50**, 3) and Chapman (*S.P.A.*, 1926, **51**, 548) have called attention to the natural presence of arsenic in fish. The effect on olive oil of keeping sardines in it has been investigated by Henseval and Deny (*Analyst*, 1904, **29**, 115); see also Bull and Saether (*J.S.C.I.*, 1910, 888).

The official reports for England and Wales give figures which show that the percentages of adulteration were as follows; the Preservative Regulations came into force in 1927:—

ADULTERATION OF FISH IN ENGLAND AND WALES

	1902-5.	1906-13.	1920-6.	1927-30.
Shrimps, fresh and potted .	35.6 %	—	—	—
Potted fish	—	36.6 %	16.4 %	0.9 %
Fish, not canned, not potted .	—	35.1 %	10.3 %	3.2 %

PROSECUTIONS FOR SARDINES IN OIL. *Wolverhampton.* The tins used were marked "In pure olive oil," and the Public Analyst found that cottonseed oil had been used, 19 % of which was present. The Government analysts were unable to make an analysis owing to the mouldy condition of the sardines. Within fourteen days information was given to the vendor that legal

proceedings were likely, but not till eighteen days more had elapsed did he take it to other analysts ; then no free oil was present, it probably having been absorbed by the sardines. The magistrate was satisfied that there was no negligence or want of care by the inspector in the method of sampling, and fined defendant £1. On appeal, *Winterbottom v. Allwood* (1915), the High Court decided that the purchaser was prejudiced, as he asked for olive oil and did not receive it. Also, that the defendant had an opportunity of having his sample analysed, and that the Court was not prepared to decide that any possibility of detriment of the sample must be prevented. The conviction was confirmed (*Grocer*, 1914, April 4, May 23 ; *B.F.J.*, 1914, 78, 99, 209).

London, Bow Street. Applying a false trade description, "Norwegian Skipper Sardines" to Norwegian brislings. "Sardine" was the French name for the pilchard, which belonged to another branch of the family "clupeidæ" to the brisling. Two vendors were each fined £20 and 100 guineas costs. On appeal to Quarter Sessions the conviction was quashed, but a further appeal to the High Court, *Lemy v. Watson and Another* (1915), confirmed the conviction (*B.F.J.*, 1914, 79, 113 ; 1915, 30, 151).

Stoke. Selling sardines packed in 100 % cottonseed oil, instead of olive oil as stated, and also applying a false trade description to them. Another Public Analyst stated the oil was olive oil, and the Government analysts found no definite evidence of the presence of cottonseed oil. Cases dismissed and the defendant allowed 20 guineas costs (*Grocer*, 1931, Nov. 14, 21 ; *B.F.J.*, 1931, 120).

PROSECUTION FOR PRAWNS. *Middlesbrough.* Boric acid 102 grains per lb. Fine £5 (*B.F.J.*, 1925, 49).

PROSECUTIONS FOR POTTED SHRIMPS. *Ormskirk.* Boric acid 41 grains per lb. in the shrimps, and 5 grains in the butter. Fine £5 (*B.F.J.*, 1902, 165).

Hull. Boric acid 176 grains per lb. Fine 5 guineas (*Grocer*, 1920, Dec. 18 ; *B.F.J.*, 1921, 9).

Liverpool. Boracic acid 2,400 parts per million. The defendant stated that he had added cayenne and mace to the shrimps as he bought them, but no preservative. The shrimps had been previously boiled on the shrimpers' boats. The stipendiary fined him £10 as he sold the preserved shrimps, though he might not have added the boracic acid. The conviction was quashed on appeal to Quarter Sessions (*Grocer*, 1931, Nov. 28 ; 1932, Jan. 16).

PROSECUTIONS FOR POTTED LOBSTER. *Blackpool.* Boric acid 61 grains per lb. Fine £1 (*Grocer*, 1905, Sept. 16).

Blackpool. Fish 65 %, bread 20 %, boracic acid 0.5 %, with some colouring ; the fish was either skate or ray. For the defendant it was argued that crayfish was river lobster. Fine £1 (*Grocer*, 1908, March 7 ; *B.F.J.*, 1908, 51).

Blackpool. Foreign fish, coloured with coal-tar dye, at least 50 %, bread 7 %, boric acid 0.2 %. The Public Analyst stated that the microscope showed that the flesh was not that of a crustacean, and that only about 25 % of lobster was present. The defendant said he had added ground rice to preserve the fish and had not used bread. Fine £3 (*B.F.J.*, 1908, 86).

PROSECUTION FOR CRAB AND LOBSTER PASTE. *Ormskirk.* Borates, expressed as boric acid 21 grains per lb.; benzoates, expressed as benzoic acid, upwards of 15 grains per lb. Salt 2.2 %, also present. Fine 10s. (*B.F.J.*, 1914, 59).

PROSECUTIONS FOR BLOATER PASTE. *Aylesbury.* Foreign ingredients—to wit, boiled starch and water 70 %. The Public Analyst said that about 30 % of herring was present, coloured with red oxide of iron. The objection to the certificate by the defence, that it did not separately state the proportions of boiled starch and water, was overruled. The Bench were asked to note that “bloater paste” was asked for and not “potted bloater.” Fine £2 (*B.F.J.*, 1901, 360).

Westminster. Boric acid 4.2 grains per lb. The glass jar was labelled: “Guaranteed pure and free from preservative.” The defendant’s analyst found no boric acid, and the Government analysts 2 grains per lb. Evidence was given that the paste was manufactured from bloaters, anchovies, American bacon and margarine. Paid costs, £10 10s. (*Grocer*, 1922, April 15; *B.F.J.*, 1922, 36).

PROSECUTION FOR POTTED SALMON. *Leeds.* Starchy filler 20 %, instead of seasoning and melted butter. It was salmon paste. Paid costs (*B.F.J.*, 1931, 68; *Analyst*, 1931, 56, 742).

PROSECUTION FOR SALMON AND ANCHOVY PASTE. *Nottingham.* Starchy filler 19.65 % and added water 26 %. The magistrates found that the quantity of added water was excessive, and inflicted a nominal fine of £1 1s. An appeal to Quarter Sessions followed, and this decision was reversed. The Recorder said that the prosecution had failed to give evidence as to what was commercially known as salmon and anchovy paste (*Grocer*, 1921, May 28, July 16; *B.F.J.*, 1921, 60, 64).

EGGS

Eggs first received the attention of the Public Analyst in war-time, when they were very scarce and dear. In 1916 a Birmingham sample described as “Complete Egg Dried” contained 77 % of protein (percentage of N \times 6.38), 5.2 % of ash, 14 % of water, and only 0.04 % of fat! It only represented the white of egg. Boric acid was absent. In the next year a tin of liquid egg marked

"Guaranteed Pure," with directions for its use for scrambled eggs and custard, contained 1.7 % of boric acid. Another sample, labelled that it had "A small percentage of boric acid," actually contained 1.5 %. Such articles had been imported from China and Japan for dressing glove leather and other industrial purposes, and had been diverted to be used as human food, without any consideration as to the injurious effects of such a large proportion of boric acid. In 1921 a sample of liquid egg used for sponge cakes contained 1.8 % of boric acid.

Four samples of dried eggs, examined about the same time, had an average composition of : protein 42.2 %, fat 38.4 %, ash 3.3 %, and water 7.9 %. One sample consisted of three packets, each of which was stated to represent one whole egg. The contents weighed 172, 162 and 128 grains, respectively, and on the average contained 65 grains of protein and 57 grains of fat. As the average weight of a hen's egg is 2 oz., and the average composition is about : protein 12 %, fat 10 % and ash 1.0 %, a whole egg contains : protein 105 grains and fat 87 grains. Each packet therefore represented only about two-thirds of an egg.

Beach, Needs and Russell have given analyses of three samples of dried egg and of one of liquid egg (*S.P.A.*, 1921, **46**, 280).

For the storage, grading and decomposition of eggs, reference should be made to Special Report No. 26 of the Department of Scientific and Industrial Research (*Analyst*, 1926, **51**, 247 ; also *Analyst*, 1930, **55**, 635). Thomson and Sorley have examined the methods of determining fat in eggs, and the results of decomposition (*S.P.A.*, 1924, **49**, 327). Hehner has noted (*Grocer*, 1908, April 25) that on keeping some of the water from the white diffuses into the yolk. Fresh eggs showed 88.0 % of water in the white, and 50.4 % in the yolk, while for lime-preserved eggs the figures were 86.0 % and 58.8 %, respectively. Nicholls has given methods for the examination of eggs suspected of being preserved (*S.P.A.*, 1931, **56**, 383).

Instead of boric acid, glycerin has been used for preserving eggs. Methods for determination of moisture, fat, and glycerin have been given by Cockburn and Love (*S.P.A.*, 1927, **52**, 143).

According to Plimmer ("Analysis and Energy Value of Foods," 1921), the average weight of hen's eggs is 55.8 gm., and the average composition : white 59.7 %, yolk 30.2 %, shell 10.1 %.

According to the Ministry of Health Reports, six of the 246 samples of dried eggs examined in England and Wales during 1920-8 were adulterated, and thirty-three of the 148 samples of liquid eggs. Of the latter, thirteen were condemned in 1922.

PROSECUTIONS FOR LIQUID EGGS. *Westminster.* Boric acid 64 grains per lb. They had been imported from China, and

the boric acid had been added to preserve them during their journey through the tropics. After six days' hearing the magistrate dismissed the summons and allowed the defence 50 guineas costs (*Grocer*, 1918, March 23, June 1).

Kensington. Boric acid 53, 51, 44 and 118 grains per lb., respectively. The Borough Council incurred costs amounting to £300 in preparing the cases. The defendants pleaded guilty to selling articles injurious to health, and were fined £15 and 35 guineas costs in each case (*B.F.J.*, 1919, 58, 66; *Grocer*, 1919, April 19, May 10).

PROSECUTIONS FOR NEW-LAID EGGS. *Reepham.* The Public Analyst said thirteen of the eighteen eggs gave evidence of the presence of water-glass, but not without analysis. It was pleaded that the eggs were of the nature and substance, but not of the quality, demanded, and that the inspector was not prejudiced, as the appearance showed they were water-glass eggs. Fine £5 (*Analyst*, 1926, 51, 141).

Glasgow. Exposure for sale after removal of the indication of origin from the eggs, and the use of a false trade description "Extra large new-laid country eggs from the Mearns." Fines £12 (*Grocer*, 1930, Nov. 1).

Bourne. Sixty dozen eggs, on each of two days, which were not of the quality demanded, not being new-laid. Evidence was given by an agricultural produce merchant, a farmer, and a sanitary inspector, that the eggs were not new-laid. Fine and costs £7 (*B.F.J.*, 1930, 106).

PROSECUTIONS FOR EGG SELF-RAISING FLOUR. *London, Lambeth.* Flour mixed with baking powder and coloured potato starch, no egg being present. The defendant stated that the article had been sold under that name for forty years, and that the class of persons who bought it did not expect to find any eggs. The summons was dismissed and the defendants allowed 2 guineas costs (*B.F.J.*, 1922, 56).

London, Lambeth. No egg or egg substance present. The summons under the Food and Drugs Act was withdrawn, with 3 guineas costs against the Council. There was an illustration of an egg on the packet, and it was said to have been intended to be sold as "egg substitute." The vendor was ordered to pay £6 5s. in fines and costs, under the Merchandise Marks Act, for applying a false trade description (*B.F.J.*, 1922, 63, 77).

PROSECUTION FOR EGG FLOUR. *Lichfield.* Contained no egg, but was self-raising flour, to which had been added a small percentage of maize, coloured with an aniline dye, which looked like eggs. It was labelled "Requires no eggs, egg powder or baking powder." Fine 10s. and £6 15s. costs (*Grocer*, 1928, Dec. 8; *Analyst*, 1929, 54, 105).

CUSTARDS. CUSTARD POWDERS

Custard is a well-known article made from eggs, milk and sugar. In 1924 nine samples of custards were bought in Birmingham and found free from boric acid. The average composition of three of them is given below ; 36 % was custard and 64 % pastry :—

ANALYSES OF CUSTARDS

	Whole Custard.	Custard part.	Pastry part.
Protein ($N \times 6.25$)	6.6	8.4	5.6
Fat	13.8	8.3	16.9
Ash	0.6	1.0	0.4
Moisture	47.0	67.9	35.2
Starch, etc.	32.0	14.4	41.9
	<hr/> 100.0 <hr/>	<hr/> 100.0 <hr/>	<hr/> 100.0 <hr/>

Some makers of custard powders have put on their products labels making great claims, such as " Makes perfect custard without eggs," " Makes the richest custard without eggs at half the cost and trouble," and " Makes extra creamy custard."

Custard powders usually contain under 1 % of protein and under 0.3 % of fat. They are composed of coloured and flavoured starch, often maize, but sago, potato or rice have been found. According to the directions, about an ounce should be added to a pint of milk. The custard powder adds only 1 or 2 grains of protein and fat to the 300 grains or so of each in the milk ; while a pint of true custard, as given above, contains about 730 grains of each. Many people like " custard " made from powder ; but let no one delude himself with the idea that its food value is that of true custard. In the case of invalids, particularly diabetic patients, the substitution of the one for the other may be serious ; the thickening is in one case due to starch and in the other to albumin.

One fraudulent label claimed " 24.37 % of proteid," while the actual amount was 4.6 %. The vendor was cautioned for the false label. Some makers label their packets honestly, as " Artificially coloured, and not made with or from eggs." Hinks (*S.P.A.*, 1923, 48, 542) has given analyses of " Real egg custard powders " which contained eggs and milk.

Of the sixty-nine Birmingham samples examined the percentage of ash in 60 % of them was 0.04–0.29 % ; in 25 % it was 0.3–0.58 % ; in 11 %, 0.6–0.99 % ; and in 4 %, 1.0–1.8 %. The high amounts of ash were due to the presence of 1.4 % of salt. Samples from two makers contained about 50 % sugar, and another make had the undesirable addition of about 0.2 % saccharin.

Of the samples of custard powder examined in England and Wales during 1919–30, 1.0 % were adulterated.

PROSECUTIONS FOR CUSTARD OR CREAM CUSTARD.

Haltwhistle. Maize flour with a little colouring and flavouring 100 %. Fine £2 (*Grocer*, 1924, Dec. 27 ; *B.F.J.*, 1925, 9).

Newcastle-on-Tyne. Cases against three manufacturers were withdrawn on the undertaking to use correct labels (*Grocer*, 1925, May 20).

PROSECUTION FOR EGG CUSTARD. *Marylebone.* Not more than 4 % of dry egg was present, equal to about 13 % of egg. The magistrate said that the amount of egg, though very small, was not, at the price paid, so small as to be to the prejudice of the purchaser (*Grocer*, 1926, March 6 ; *Analyst*, 1926, 51, 188 ; *B.F.J.*, 1926, 35).

PROSECUTION FOR CUSTARD POWDER. *Bradford.* Not of the substance and quality demanded. One vendor was fined £3 and another £1 (*Grocer*, 1919, Jan. 18).

CHEESE

The Report of the Food Products Adulteration Committee (1896) mentioned imitation cheese, known as filled cheese, oleine cheese, or margarine cheese, and also cheese made from skimmed milk. The 1899 Act required margarine cheese to be marked in a similar way to margarine ; it defined cheese as "the substance usually known as cheese, containing no fat derived otherwise than from milk," and gave power to the Board of Agriculture to make regulations as to the composition of cheese.

The Board, in answer to requests that regulations should be made, declined, considering that, while it might be practicable to fix a limit for fat, it was doubtful if any useful purpose would be served. Such limits are common abroad, and have been summarised by Swaving (*Analyst*, 1924, 49, 230).

The richness of cheese may be expressed in two ways—(1) as the percentage of fat in the cheese itself, or (2) as the percentage of fat in the water-free cheese. The latter will depend only on the composition of the milk used, but the former will also vary with the amount of water in the cheese.

In the manufacture of cheese, 1,000 lb. (nearly 100 gallons) of milk will yield about 100 lb. of cheese, and as about 0.3 % of fat remains in the whey, cheese made from average whole milk, containing 3.6 % of fat, will have about 33 % of fat. Richmond (*S.P.A.*, 1919, 44, 202) has given a formula for calculating the composition of the milk used in making a cheese.

As whole milk contains more fat than casein, and as most of the lactose remains in the whey, at least half of the dry matter of such cheese should be fat. Various published official figures show the reasonableness of this expectation. In 1914, 327 samples of Cheddar and Caerphilly cheeses had 47.1–56.8 %, with an average of 51.3 %

of fat in the water-free cheese. Of Canadian cheese 444 samples had an average of 51.8 % fat in the dry cheese. Only twenty-seven of the 453 samples of "cheese" examined by the Lancashire County Analyst, 1926-30, had less than 45 % of fat in the dry cheese (1930 Report).

Graded Cheshire cheese is guaranteed to contain 46-50 %, and "standardised" New Zealand cheese 50-52 %, although some fat has been removed from the milk used. Cribb (*S.P.A.*, 1906, **31**, 105) and van Rijn (*S.P.A.*, 1915, **40**, 391) have given analyses of Dutch cheese; the latter states that whole-milk cheese with less than 46 % of fat in the dry matter is rarely found, and that the average is about 50 %. Dutch cheese is branded 40, 30, or 20, according to the percentage of fat.

The proposal in the Sale of Cheese Bill, 1930 (*Grocer*, 1930, July 12), that cheese of which the dry matter contains less than 45 % shall be marked "Skim-milk cheese" does not appear unreasonable. The Society of Public Analysts has suggested that cheese made from skimmed milk should be marked "Skim-milk cheese" as well as "one-quarter fat," "half-fat" or "three-quarter fat," according to its quality. Also, that the legal minimum of fat in "cheese" should be 45 % of milk-fat in the dry substance (*S.P.A.*, 1931, **56**, 220).

Analyses of skim-milk cheese made by De Kruffyff (quoted *Analyst*, 1915, **40**, 396) indicate that an increase of 0.1 % of fat in the milk, produced, on the average, an increase of 1.75 % of fat in the water-free cheese; 0.5 % yielding 12.45 %, and 2.0 % yielding 38.65 %.

Not only are there no official limits for "cheese," but also none have been fixed for particular varieties, such as "Cheshire cheese," and prosecutions have been hindered by this fact. The incorrect statement has been made that magistrates have no power to fix limits.

During 1921-7 the amount of water was determined in 814 samples bought as "cheese" in Birmingham, with the following results:—

WATER IN "CHEESE," 1921-7 (814 samples)

Percentage of water	. 21.4	26	30	34	36	38-41.8	Total.
Percentage of samples	. 6	42	43	6	2	1	100

The average of the ashes of forty-four samples examined in 1927 was about 4.1 %; the extreme figures were 3.1 % and 4.8 %. The B.-R. 40° of the fat were rarely outside the limits 42-45 (R.I. 1.4538-59).

REICHERT VALUES OF "CHEESE," 1921-9 (808 samples)

Reichert value	. 21.9	24	26	28	30	32-33.9	Total.
Percentage of samples	. 1	6	23	33	27	10	100

The Reichert results are a striking contrast with those obtained with the fats of margarine cheese, which were 0.2–2.6. Lowe (*S.P.A.*, 1928, **53**, 89) has called attention to the low Reichert-Meißl figure (22.9) which may be obtained with the fat from cheese made from the milk of cows advanced in lactation.

Two samples bought in Birmingham as “Cheshire cheese” had 40.8 % and 27.7 % of fat respectively; twenty-two others had 29.1–37.8 %. Ten samples bought as “Cheddar cheese” had 31.6–44.4 % of fat, 21.8–36.2 % of water, and the percentages of fat in the water-free matter varied 43.6–60.9 %. B.-R. 40° C. of the fat of these thirty-four cheeses were 43–46 (1.4545–1.4566).

Of the samples of “cheese” bought in England and Wales during 1888–1902, 2.2 % were adulterated. During 1903–13, the proportion was 1.6 %, and during 1919–30, 2.3 %.

Gorgonzola Cheese. In 1910, three of the thirteen Birmingham samples examined had been heavily coated with a rind, about $\frac{1}{8}$ -inch thick, of barytes and tallow, tinted with oxide of iron. In these samples the rind was 10.6–14.0 % of the total cheese, and yielded 71–83 % of ash, which was equal to 7.5–11.5 % of ash on the total cheese. The ash of the rind of the other samples was only 0.3–0.7 % of the total cheese. The sale of coated cheese was again reported in 1930, but six Birmingham samples did not contain more than 1.4 % of ash of rind expressed on the total cheese (cp. Hinks, *S.P.A.*, 1911, **36**, 61).

The proportion of fat in the interior part of eighteen of twenty-four samples examined varied 26.5–36.7 %, while extreme figures were 21.7 % and 42.8 %. Nine out of eleven samples had 33.2–41.8 % of water, other figures being 42.8 % and 49.6 %. The fat in nine out of eleven samples was 44.5–57.9 % of the water-free matter, the other two samples having 38.0 % and 39.6 %.

Cream Cheese. There has been gross misuse of this name, samples sold as “cream cheese” having from 1.2–76 % of fat! It appears obvious to the writer that a person buying “cream cheese” does not expect one made from skimmed milk, nor one made from whole milk, but a cheese considerably richer in fat than either. The Sale of Cheese Bill, 1930, suggests a limit of at least 80 % of fat in the dry matter. Analyses have been given by Cribb (*S.P.A.*, 1909, **34**, 45) and Hodgson (*S.P.A.*, 1924, **49**, 264); the latter has emphasised the importance of considering the relation between the protein and fat contents of the cheese. Sometimes “Bondon cheese” is incorrectly sold as cream cheese, it being made from whole milk or skimmed milk (see Elsdon, *S.P.A.*, 1924, **49**, 267).

Metallic Impurities. Stoddart reported the presence of metallic lead in cheese (*S.P.A.*, 1897, **22**, 2); Allen and Cox mentioned the use of zinc sulphate as “cheese spice” (*S.P.A.*, 1897, **22**, 187);

Elten found 2.3 % of tin in the surface of rindless cheese in contact with tin-foil that was discoloured (*Analyst*, 1929, **54**, 552) and Manley tin and antimony in Gruyère cheese that had been packed in metal foil (*S.P.A.*, 1930, **55**, 191); he also gave a method for analysis (see also *B.F.J.*, 1931, 1; *Analyst*, 1931, **56**, 251). The darkening, which increases with time, is partly due to the acidity of the cheese, and can be much decreased by the use of coated tin-foil (Ottiker, *Grocer*, 1930, June 14).

ANALYSIS. *Moisture.* Weigh about 2 gm. in thin slices in metal dish with flat bottom. Dry in water oven three hours, weigh, and heat another hour.

Determination of Fat. Boil about 3 gm. in thin slices in a wide test tube with 5 ml. of water and 10 ml. 10E.HCl with constant shaking till all but fat is dissolved. Transfer hot to separator, cool and shake out with four quantities of ether. Distil off ether, dry, weigh and determine refraction. Direct extraction of cheese with ether may give low results (Cribb, *S.P.A.*, 1906, **31**, 111).

Separation of Fat. Cut up into thin slices, put in enamel funnel, with 11 cm. filter paper over the holes of the funnel. Support the funnel in a beaker, and heat under water oven. Overheating may hinder the separation of the fat. Prolonged heating (fourteen hours) lowers the Valenta figure, but has little effect on the refraction of the fat.

Nitrogen. Determine on 2 gm. Multiplication of nitrogen by 6.38 gives percentage of casein, etc.

Chlorides. Cornish and Golding have devised a method (*S.P.A.*, 1915, **40**, 197).

PROSECUTIONS FOR CHEESE — EXCESS WATER, DEFICIENCY OF FAT. *Liverpool.* Water 47.4 %, instead of 25–35 %. The defendant's analyst found 38 %, and the Government analysts 41 %. Withdrawn on payment of costs (*F. & S.*, 1898, July 9).

Wolverhampton. Butter-fat 12.3 %, water 40 %. It was Dutch skim-milk cheese. The stipendiary dismissed the case, considering that as the fat was derived from milk, it came within the definition of the Act, and as the Board of Agriculture had not fixed a standard (*B.F.J.*, 1900, 147, 165).

Kensington. Fat 9.6 %, being 40 % deficient of the amount that should have been present. The analyst suggested that 16 % was a very reasonable and low limit. The defendant said he bought it as "Kosher cheese." Nominal fine £1 (*Grocer*, 1906, Jan. 20; *B.F.J.*, 1906, 48).

West Hartlepool. Fat 7 %, water 52 %. It was argued that the Bench had no right to make a standard. It was a Dutch cheese (Gouda) and was sold at a cheap price. Dismissed (*Grocer*, 1920, June 5; *B.F.J.*, 1920, 55).

PROSECUTIONS FOR MARGARINE CHEESE. *Birmingham.* Exposure for sale, unmarked. The assistant was fined 10s. and the employer was ordered to pay costs (*B.F.J.*, 1900, 164).

PROSECUTIONS FOR FOREIGN FAT IN CHEESE. *Liverpool.* Lard and beef fats 14 %. The manufacturers were said to have been making such cheese for eight years. Fine £5 (*F. & S.*, 1892, Nov. 5).

Prescot. Lard 22 %. Fine £2 (*F. & S.*, 1895, Sept. 28).

Birmingham. Foreign fat 30 %. Fine £5 (*F. & S.*, 1897, Oct. 9).

Hastings. Fat not derived from milk 20 %. The Government analysts found only milk fat. The case was withdrawn and the defendants allowed 10 guineas costs (*Grocer*, 1904, Oct. 1, 29).

Bunff. Margarine cheese. Objections were taken to the certificate that it did not state the amount of foreign matter, and if any change had taken place. The sheriff considered the cheese was not adulterated but a well-recognised commodity, and that it could be kept for a long period without change. Fine £1 (*Grocer*, 1910, March 19, 26; *B.F.J.*, 1910, 54, 70).

PROSECUTIONS FOR CHEDDAR CHEESE. *Burnley.* Fat 15.5 %, instead of at least 25 %. It was sold as "Dutch Cheddar." Fine 10s. (*Grocer*, 1929, May 4; *B.F.J.*, 1929, 65).

Aylesbury. Obtaining money under false pretences by sale of margarine cheese as Cheddar. A piece of Cheddar cheese had been inserted into the top of the cheese. The sentence was nine months' imprisonment (*Grocer*, 1929, Dec. 28; *Analyst*, 1930, 55, 127).

PROSECUTIONS FOR CHESHIRE CHEESE. *Nottingham.* Fat 18 %, instead of a minimum of 30 %. Fine £5 (*Grocer*, 1908, April 4, 18, May 23; *B.F.J.*, 1908, 65, 103).

Knutsford. Fat 14 %, being 30 % deficient of the lowest standard of 20 %. The magistrates decided that any cheese which contained less than 20 % of fat ought not to be entitled to the prefix "Cheshire." Fine 5s. (*Grocer*, 1911, Feb. 4; *B.F.J.*, 1911, 30).

Stockton. Fat 17 %, instead of at least 24 %. From the evidence the magistrates were of opinion that Cheshire cheese must be made from practically whole milk, and contain at least 24 % of fat. Fine £1 (*Grocer*, 1914, June 13; *B.F.J.*, 1914, 116).

Salford. Fat deficient 26 %. It was stated that the genuine article never contained less than 45 % of fat in the water-free cheese, and that the cheese itself had 28-30 % of fat in it. It was Dutch cheese. Fine £2 (*Grocer*, 1924, March 22; *B.F.J.*, 1924, 35).

Stoke-on-Trent. Fat calculated on the moisture-free substance 37.7 %, instead of 45 %, as recognised by the Cheshire Farmers' Union. Fine £1 (*Analyst*, 1927, 52, 151; *Grocer*, 1927, Jan. 22).

PROSECUTIONS FOR DUTCH CHEESE. *Coventry.* Fat 14.65 %, being 50 % deficient. It was "Gouda" cheese. Fine 1s.

An appeal to Quarter Sessions allowed. The deputy-chairman stated that as the evidence indicated a variation from 15 % to 25 % or 30 %, they were unable to fix a standard, and the appeal was allowed, with costs (*B.F.J.*, 1903, 89, 159).

Wokingham. Fat 1.6 %, water 57 %. The Public Analyst stated that genuine Dutch cheese should contain 20–25 % fat, and not more than 40 % water. At the time of sale in the Market-place, the defendant was shouting “This is the best cheese in the kingdom.” Fine £10 (*B.F.J.*, 1903, 164).

London, Marlborough Street. Fat 2.8 %, instead of 16 %. The magistrate dismissed the case, considering the purchaser asked for Dutch cheese and got it, and allowed 20 guineas costs (*Grocer*, 1905, March 25, April 1, May 20).

Cambridge. Fat 10.83 %, instead of 16 %. For the defence, it was argued that the Board of Agriculture had refused to fix a standard and that the limited experience of the Public Analyst was not sufficient for the purpose ; also, that while admitting the article was liable to decomposition, the Analyst had made no reference to it on his certificate. Dismissed (*Grocer*, 1912, March 23 ; *B.F.J.*, 1912, 54).

PROSECUTIONS FOR GORGONZOLA CHEESE. *Kingston-on-Thames*. Substances foreign to cheese 14.6 %, consisting of a rind composed of 15 % of tallow and 85 % of barytes coloured with oxide of iron. Evidence was given that the cheese beneath the coating was slimy and unpleasant. The defence admitted that a 20-lb. cheese had a 4-lb. coating. Fine £10. A notice of appeal to Quarter Sessions was given, and afterwards withdrawn (*Grocer*, 1910, Oct. 1 ; *B.F.J.*, 1910, 194).

Lambeth. Barytes 10.2 %, tallow 3.2 %. The magistrate dismissed the case as the coating had no effect on the cheese, but was put on to preserve it (*Grocer*, 1910, Nov. 26 ; *B.F.J.*, 1910, 236).

Kingston. Artificial rind containing 79 % of barytes. Fine £5 (*Grocer*, 1930, Feb. 1 ; Analyst, 1930, 55, 194 ; *B.F.J.*, 1930, 26).

PROSECUTION FOR MILK CHEESE. *Salford*. Fat 2.0 %, or 7.3 % calculated on the water-free cheese, instead of at least 45 %. It was labelled “Bondon Milk Cheese,” and was said to be made from separated milk with the addition of a little whole milk. The makers were fined 10s. 6d. and 5 guineas costs for issuing a false label (*Grocer*, 1929, June 22 ; Analyst, 1929, 54, 540 ; *B.F.J.*, 1929, 85).

PROSECUTIONS FOR CREAM CHEESE. *Burnley*. Fat 25.6 %, 7.5 % and 5 %, respectively. The first case was dismissed and the defendants in the two later cases were fined 10s. each (*Grocer*, 1911, Jan. 7).

Kensington. Boric acid 36.07 grains per lb. The Government

analysts subsequently found 36.4 grains per lb. Fine £10 (*B.F.J.*, 1916, 407, 460).

Kensington. Fat 1.95 %, instead of the very low standard of 16 %. Fine £5 (*B.F.J.*, 1916, 437).

Wallasey. Fat 6.5 %, instead of 30 %. It was a soft cheese made of skimmed milk. Fine £1 (*Grocer*, 1925, Sept. 25; *B.F.J.*, 1925, 104; *Analyst*, 1925, 50, 553).

PROSECUTION FOR CREAM CHESHIRE CHEESE. *Salford*. Fat 32 % instead of 70 %. It was a whole-milk cheese, and not a cream cheese. Fine £10 and 10 guineas costs (*Grocer*, 1925, June 13; *Analyst*, 1925, 50, 402; *B.F.J.*, 1925, 66).

PROSECUTION FOR CHEESE SANDWICHES. *North London*. Fat, other than milk fat, 67 %, in the cheese of the sandwiches. Paid 4 guineas costs (*Grocer*, 1931, May 2; *Analyst*, 1931, 56, 399).

North London. Fat, other than milk fat, 85 %, in the cheese of the sandwiches. It was stated that it was necessary to use margarine or the article would be unsaleable in a few weeks. The vendor proved a warranty, and the manufacturer was ordered to pay 10 guineas costs for giving a false warranty (*Grocer*, 1931, May 16, 23, July 25; *B.F.J.*, 1931, 57, 64, 75).

CHAPTER XXI

FRESH AND PRESERVED FRUITS, SEEDS, Etc.

Apples. Oranges. Dried fruit, raisins, sultanas, currants. Candied fruit. Ground almonds. Lentils. Peas. Beans. Spinach. Capers.

PROSECUTIONS for these foods may be divided into three main classes :—(1) Those due to the improper or excessive use of preservatives—boric acid and sulphur dioxide. (2) Those due to the methods of preparation—arsenic, lead and zinc. (3) Those for copper, used to preserve the colour of green peas, etc.

Apart from these the sale of “ articles, not of the nature and substance of the article demanded ” are rare. The foods usually have obvious characters well known to buyers, and any substitution is probably accidental, as when lentils were supplied for pearl barley.

Macara has given analyses of a large number of fruits which are used for making jam (*S.P.A.*, 1931, **56**, 39).

The following percentages of adulteration refer to England and Wales :—Fruits in syrup, 1905–13, 15·3 % ; bottled and tinned fruit, 1920–30, 4·4 % ; preserved vegetables, not peas, 1906–10, 42·4 % ; preserved vegetables, including peas, 1922–7, 30·3 %, and 1928–30, 0·9 %.

APPLES

In December, 1915, a circular (No. 659) was sent out by the Ministry of Health calling attention to the importation of Jonathan apples containing up to $\frac{1}{10}$ grain arsenic per lb., and stating that two cases of arsenical poisoning had followed their consumption.

Cox (*S.P.A.*, 1926, **51**, 132) found that two suspected samples of these apples contained 8 parts of arsenic and 28 parts of lead per million, and that other samples of American apples contained 0·5–15 parts per million. He stated that, in order to prevent the ravages of the codlin moth, apples were sprayed with a solution of lead arsenate, and that washing would not entirely remove the arsenic from the skin. Swingle has given the composition of lead arsenate, and its effect on peach trees (*Analyst*, 1930, **55**, 60).

Caldwell (*Daily Mail*, 1926, Feb. 11) found that the powdery residue in the hollow round the stalk contained a dangerous proportion of arsenic, and that it was also present in the core.

In his Report for 1926 (*Analyst*, 1928, **53**, 287) the Dominion Analyst for New Zealand gave the composition of a typical spray,

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which was applied several times in the course of the season. In only two cases did the arsenic exceed $\frac{1}{100}$ grain per lb., and these were examined within a week of spraying. Arsenic in the pared fruit was negligible as compared with that in the skin. Wiping the skin was found to be of little value, except for patches of visible spray. See also Shutt (*S.P.A.*, 1926, **51**, 291), Barnes (*Analyst*, 1929, **54**, 347), and Henderson (*Analyst*, 1929, **54**, 747). Lendrich and Mayer give a method for determination of arsenic and lead (*Analyst*, 1927, **52**, 237).

ARSENIC IN APPLES, 1925-30

Grain arsenic, per lb.	75	$\frac{1}{100}$	$\frac{1}{250}$	$\frac{1}{150}$	$\frac{1}{90}$	$\frac{1}{1000}$	and less.	Total.
No. of samples	1	2	15	9	68			95

PROSECUTIONS. *Hampstead.* Arsenic $\frac{1}{5}$ and $\frac{1}{30}$ grain per lb., respectively. They were bought owing to a case of illness due to eating American apples. Each vendor was fined £1 (*Grocer*, 1925, Nov. 28 ; *B.F.J.*, 1925, 113).

London, Bow Street. Arsenic $\frac{1}{4}$ grain per lb. The certificate was challenged as not giving sufficient information, but after consideration, the magistrate accepted it and ordered the vendor to pay £5 costs (*Grocer*, 1926, June 5, 12 ; *B.F.J.*, 1926, 54, 66).

Feltham. Arsenic 0.05, and lead 0.11, grain per lb. The apples were stated to have been cleaned with a damp cloth. The Public Analyst observed a slight deposit on the calyx and stalk. Fine 19s. (*Grocer*, 1926, April 8 ; *B.F.J.*, 1926, 44).

Marylebone. Arsenic $\frac{1}{2}$ grain per lb. Proceedings were the direct result of a complaint that people were ill after eating apples. Fine £10 (*B.F.J.*, 1926, 34).

ORANGES

In 1924 Birmingham Market inspectors noticed oranges on the barrows of hawkers which appeared to be ripe, while no ripe oranges were obtainable in the wholesale market. Investigations followed, and it was found that one of the hawkers dyed the unripe oranges with chrysoidine, which is a dye used to make bait more alluring to fish. The calyx of an orange is green, but that of a dyed one is red. The colour was detected by boiling the peel with water containing 10 % of potassium bisulphite and woollen fabric (nun's veiling). Oranges colour the fabric pale yellow, and dyed oranges a deep yellow (*S.P.A.*, 1925, **50**, 183). Although the practice is objectionable, it can hardly be called adulteration ; chrysoidine is not a prohibited colour.

In 1930 similar oranges were seen on a barrow marked "Ripe juicy oranges," and if they had been bought as such, they would

not have been of the "quality" demanded. Unfortunately, when they were bought later, the ticket had been changed to "Murcia oranges," which was a more difficult thing to disprove. See also Pum and Micko (*Analyst*, 1901, **26**, 73). Grape fruit has been similarly dyed in America, and an order was given for its destruction (*Analyst*, 1926, **51**, 186).

Dunn and Bloxam (*S.P.A.*, 1929, **54**, 28) detected boric acid in oranges, the average amount in the peel of seven samples being 1.12 grains per lb., and in the pulp 0.28 grain.

DRIED FRUIT

The L.G.B. Report for 1913 called attention to the presence of sulphur dioxide in dried fruit, seven out of twenty-six samples containing over 4 grains per lb. Dried apricots contained 2.2-7 grains. The Final Report of the Preservative Committee, 1924, gave particulars of the application of sulphur dioxide to apricots and peaches, and stated that currants, raisins, figs, prunes, apples, and pears are either not sulphured, or only lightly treated. The Preservative Regulations fixed a limit of 2,000 parts per million for apricots, peaches, nectarines, apples and pears. Miller (*S.P.A.*, 1927, **52**, 338) found 220 (apple rings) to 1,580 (peaches) parts of sulphur dioxide per million. Of the sixteen samples of dried fruits examined in Birmingham in 1929 fifteen had not more than traces, and one of dried apricots had 410 parts sulphur dioxide per million; all were free from boric acid. Experiments by Beythien and Bohrisch (*Analyst*, 1902, **27**, 226) and Cooksey (*Analyst*, 1928, **53**, 540) proved that about half of the sulphur dioxide remained after cooking.

Scott Dodd (*S.P.A.*, 1929, **54**, 16) detected traces of boric acid in dried fruits, varying from 40 (prunes) to 300 (apricots and peaches) parts per million.

About 1897, Wiley pointed out that American apples dried on zinc or galvanised iron trays might contain $\frac{2}{3}$ grain zinc oxide per lb. (*F. & S.*, 1897, April 24).

Of the samples of dried fruit examined in England and Wales during 1920-30, 1.4 % were adulterated.

RAISINS, SULTANAS, CURRANTS

The Preservatives Regulations allow 750 parts of sulphur dioxide per million in raisins and sultanas. There is no allowance for currants; the Greek Minister has stated that 60,000 tons of currants are imported each year from Greece, and that not 1 oz. of preservative or colouring matter is employed (*Grocer*, 1926, Dec. 18). Fifteen Birmingham samples were free from it; they yielded 1.7-2.2 % of ash.

In 1928, three samples of sultanas were bought in Birmingham : the palest, and the dearest, contained 360 parts of sulphur dioxide per million ; the other two were free from it. This illustrates the point that the consumer had to pay for having his fruit deteriorated by bleaching. Three samples of raisins contained 0, 100 and 130 parts of sulphur dioxide per million.

Scott Dodd (*S.P.A.*, 1929, **54**, 16) detected traces of boric acid in raisins, sultanas and currants, varying from 110 to 260 parts per million.

PROSECUTIONS FOR RAISINS. *London, South-Western.* Sulphur dioxide 1,060 parts per million. The article was said to have been sold as it was received. Vendor ordered to pay £1 1s. costs (*Grocer*, 1930, Jan. 4 ; *B.F.J.*, 1930, 20).

Leicester. At least 370 parts of sulphur dioxide per million in excess of the 750 parts allowed. The sulphite was stated to have been sprinkled over the goods just before Christmas. Defendant proved a warranty and the case was dismissed (*B.F.J.*, 1930, 28 ; *Grocer*, 1930, Feb. 1).

Cardiff. Sulphur dioxide 850 parts per million. Fine £5 (*Grocer*, 1931, Feb. 7).

PROSECUTIONS FOR SULTANAS. *London, Old Street.* Currants 12·8 %. The admixture was due to carelessness. The defendant paid £2 2s. costs (*Grocer*, 1928, July 11).

Greenwich. Sulphur dioxide 990 parts per million. Paid 5 guineas costs (*Grocer*, 1932, Feb. 6 ; *B.F.J.*, 1932, 30).

CANDIED FRUIT

The Preservative Regulations allow 100 parts of sulphur dioxide per million in crystallised glacé or cured fruit, including candied peel. None of the seventeen samples of lemon, orange, or citron peel examined in Birmingham in 1928–30 contained sulphur dioxide.

“Candied skins” have been imported from which most of the essential oil has been removed before candying. They may be detected by their very deficient flavour.

Three samples of glacé cherries analysed in 1927 also contained no sulphur dioxide. Boron was present equal to about 0·3 grain per lb. of boric acid. The moisture was 17·6–18·0 %, and the ash 0·27–0·32 %.

PROSECUTION FOR CANDIED PEEL. *London, Old Street.* Sulphur dioxide 150 parts per million. A practical confirmation was given by the Government analysts. Vendor ordered to pay £3 3s. costs (*Grocer*, 1928, March 3 ; *B.F.J.*, 1928, 24).

Cardiff. Sulphur dioxide in excess 40 parts per million. The objection that the summons stated “candied peel,” while the article

was entirely lemon peel, was overruled. Fine £2 (*B.F.J.*, 1931, 14; *Analyst*, 1931, **56**, 181).

GROUND ALMONDS

In some prosecutions for the presence of 5 % of starchy matter in ground almonds it has been argued that without such addition a pulpy mass will result. The presence of starch, however, is unusual, and a grinder has stated (*Grocer*, 1923, Sept. 15) that no addition is required if the almonds are properly prepared and ground.

Below are given analyses of ground almonds, and for comparison almonds after removal of the skin, and ground arachis.

ANALYSES OF ALMONDS AND ARACHIS

Percentages.	Ash.	Protein.	Oil.	Moisture.	B.R. of Oil.	
Ground arachis . . .	2.03	27.4	46.6	—	68	at 25°
Almonds . . .	2.98	27.5	56.1	5.0	56	at 40°
Ground almonds (A) .	2.72-3.15	21.5-24.8	56.2-59.9	—	64-66	at 25°
„ „ (B) .	2.9	—	59.5	3.9	60.2	at 35°

The ground almonds marked (A) were eight samples bought in Birmingham, 1914-5, and those marked (B) were sixteen samples bought in Bristol, 1921-2 (Public Analyst's Reports). In none of these samples was any adulterant detected. The iodine values of the Bristol samples averaged 100.5 %.

The ether-extracted residue of the ground almonds was free from starch, but a notable amount was present in the ground arachis. The samples should be tasted to ascertain that there has not been accidental substitution of bitter almonds for sweet ones. The extracted oil may be tested for the kernels of apricots and peaches by Bieber's test and by nitric acid (see Fendler, Frank and Stüber, *Analyst*, 1910, **35**, 248).

PROSECUTIONS. *Darlington.* Crystallised sugar 15 %. Fine 10s. (*Grocer*, 1908, Jan. 4).

Glasgow. Starch derived from oatmeal 5 %, equivalent to 9 % of oatmeal. The Government analysts found starch equivalent to about 5 % of oatmeal. After contradictory evidence from grinders, the sheriff dismissed the case, holding that it was reasonably necessary to use an absorbent in the grinding process (*Grocer*, 1908, March 7, April 4; *B.F.J.*, 1908, 67, 69).

Wigan. Ground earth-nuts 10 % and ground rice 10 %. Fine £5. The same penalty was inflicted on another vendor who sold a sample containing 15 % of ground rice (*B.F.J.*, 1914, 127).

Nelson. Wheat flour, not less than 20 %. Fine, with costs, £5 (*B.F.J.*, 1916, 336).

Ashton-under-Lyme. Foreign cereal corresponding in character to that of barley 5 %. Fine 10s. (*Grocer*, 1923, Sept. 8; *Analyst*, 1923, **48**, 545; *B.F.J.*, 1923, 87).

Liverpool. Application of the false trade description, "Pure, sweet English ground almonds" to an article which was ground apricot kernels. Fine £20 and £90 6s. costs (*Grocer*, 1926, Feb. 20, March 6; *Analyst*, 1926, **51**, 187; *B.F.J.*, 1926, 36).

LENTILS

Analyses of lentils have been given by Balland (*Analyst*, 1897, **22**, 323). In 1905 the Warrington Public Analyst examined a sample of lentils faced with kaolin to the extent of 33 grains per lb.

The ash of twenty-four Birmingham samples varied from 1.80–2.20 %, and the ash insoluble in acid from 0.01 % to 0.44 %. Some of the samples contained grit, but the amount picked out (0.2 %) was not enough to be called adulteration.

PROSECUTION. *Banbury.* Split peas 45½ %, the remainder being lentils. Fine £5 (*Grocer*, 1929, Feb. 2).

PEAS

Analyses of peas have been given by Balland (*Analyst*, 1897, **22**, 323). Eleven Birmingham samples of dried peas differed little in ash, the variation being 2.4–2.8 %, the part insoluble in acid being 0.19–0.34 %.

To fix and intensify the green colour, peas have been treated with a solution of copper sulphate, and then washed with water to remove any excess. As the copper enters into combination with the organic matter of the peas, the presence of more than a trace of copper *sulphate* is improbable, and its presence has often been denied.

One of the ten Birmingham samples of preserved peas examined 1907–16 and three samples examined 1928–9 were free from copper. The other nine contained 0.4–1.0 grain of copper, in combination, per lb. of strained peas. The liquids drained from the peas were 31–46 % of the samples and contained 0.05–0.2 grain of copper per lb. In only four instances was the presence of copper declared on the label.

The addition of copper compounds to colour peas, etc., was prohibited by the Public Health (Preservatives, etc., in Food) Regulations, 1925, and its necessity is shown by the following summary :—

REPORTED PROSECUTIONS FOR COPPER IN PEAS

Copper, expressed as sulphate,						
grains per lb.
Number of prosecutions	.	.	45	45	21	8
						119

Many of the prosecution certificates ignored the presence of the

liquid, and in one case, at least, the inspector threw it away. It is suggested that both proceedings were illegal. The question of sampling and certifying preserved peas has been previously discussed (pp. 21, 38), and also the methods for copper (p. 87).

In green preserved peas free from copper, Cribb and Still (*S.P.A.*, 1925, 50, 286) have found as much as 3.1 grains of zinc per lb. Henville has reported finding methylene blue in tinned peas (*S.P.A.*, 1930, 55, 629; 1931, 56, 742).

It may be pointed out that there are two varieties of canned peas:—(1) Peas harvested July–August, and canned the same day. These have a superior flavour and alone are entitled to the “National Mark.” (2) A cheaper variety made from sun-dried peas, canned after soaking, by the same process. (See correspondence in *Grocer*, 1931, Feb. 21, 28, March 7, 14, 21.) Apparently the distinction between the two varieties is not always made clear on the labels, and substitution of the processed peas for the fresh peas would constitute adulteration.

Of the samples of preserved peas examined in England and Wales, 1891–1904, 52 % were reported adulterated; during 1905–13, 45 %; and from 1920–1, 40 %. All but one of the 451 samples of pea flour examined 1920–30 were genuine.

PROSECUTION FOR DRIED GREEN PEAS. *Leeds.* “A small quantity of copper sulphate.” The case was dismissed under the Probation Act. It was afterwards admitted that the proportion of copper was no more than might have occurred naturally (10–14 per million (?)) (*Grocer*, 1927, May 28, July 2).

PROSECUTIONS FOR PRESERVED PEAS. *London, Marlborough Street.* Copper equivalent to $2\frac{1}{2}$ grains of sulphate per lb. tin. Fine 1s. and 5 guineas costs (*Analyst*, 1877, 1, 216).

Kensington. Copper 0.00924 %, equivalent to 2.55 grains crystallised copper sulphate per lb., to the prejudice of the purchaser. Eminent medical evidence was given that the eating of such peas was likely to cause injury. No evidence was given for the defence, but the Bench dismissed the case. The magistrates, in their statement of appeal to the High Court, *Friend v. Mapp* (1904), said that within their own knowledge preserved peas contained a small amount of added colouring matter for preserving the natural green colour. They found that the quantity of copper present was not sufficient to render the peas injurious to health, and that the appellant received what was usually sold as “preserved peas.” The Judges dismissed the appeal, being unable to say that the magistrates had gone wrong in law, but they hinted that they themselves might have come to a different decision on the evidence. The Lord Chief Justice considered that in such cases it would be better to prosecute for the sale of an article injurious to health (*B.F.J.*, 1904, 65, 140).

Bezhill. Wilful sale of bottled peas "mixed with . . . sulphate of copper, which ingredient was injurious to health." At least 1.87 grains per lb. were present, and the Public Analyst stated that he suffered from colic if he ate coppered peas. The defendant was fined £5 and appealed—*Hull v. Horsnell* (1904). The Lord Chief Justice stated that to constitute an offence it was necessary that the food itself should be injurious to health, and not merely an ingredient. Also, that it was not necessary that the Public Analyst's certificate should have stated "which rendered the article injurious to health" (*B.F.J.*, 1904, 233; 1905, 40).

Hull. Metallic tin in solution 2 grains per lb. and copper, calculated as anhydrous sulphate, 1.03 grains per lb. The defendant's analyst found $\frac{3}{4}$ and $\frac{1}{2}$ grain per lb., respectively, and the Government analysts 1.19 of tin, and the equivalent of 1.66 grains per lb. of anhydrous copper sulphate. It was maintained that the presence of tin was not an offence, as the Act said "No person shall mix," and the tin was due to solution, and not to mixing. After conflicting evidence, the Court ordered the defendant to pay the costs of the analysis only (*Grocer*, 1910, Jan. 29; *B.F.J.*, 1910, 37).

Bridlington. Tin (metallic) 1.4 grains, and copper sulphate (anhydrous) 3.2 grains per lb. Fine £1 (*Grocer*, 1910, Feb. 5).

Kensington. "Selling peas which had been rendered injurious to health by reason of the fact that the same contained 1.656 grains of crystallised copper sulphate per lb." The case was dismissed, as the summons did not follow the Public Analyst's certificate, and he admitted that the copper was not in the form of sulphate (*Grocer*, 1911, May 20).

London, Bow Street. Copper equivalent to 3.038 grains per lb. of sulphate, and being injurious to health. The tin bore the statement the peas contained only a small quantity of copper sulphate. The Medical Officer of Health stated that in cases he had investigated three of the eight persons who had eaten coppered peas were upset, the quantity being $2\frac{1}{2}$ or 3 grains per lb. The copper present was more than twice the quantity necessary to retain the colour of the peas, and he was satisfied that a much smaller quantity than that in the sample would be injurious to the health of most consumers. Fine £21 and 10 guineas costs (*Grocer*, 1923, March 24; *B.F.J.*, 1923, 58).

Dunstable. Copper equivalent to 2.765 grains of sulphate per lb., and injurious to health. The Medical Officer of Health stated that experiments showed that 11–33 % of the copper taken into the body was stored in the liver, to its injury. Experiments made by a medical man called for the defence showed that 60 % of the copper remained insoluble when the peas were eaten. He considered the amount absorbed would not affect a person, even if suffering

from kidney disease. Fine £10 (*Grocer*, 1923, June 23 ; *Analyst*, 1923, 48, 446 ; *B.F.J.*, 1923, 68).

BEANS

Analyses of haricot beans have been given by Balland (*Analyst*, 1897, 22, 323). The ash of nineteen Birmingham samples varied from 3.05 % to 3.94 % and the ash insoluble in acid from 0.18 % to 0.47 %. Four samples of preserved beans contained 0.1–0.4 grain of copper per lb. ; only the highest had a label indicating the presence of copper.

PROSECUTIONS FOR PRESERVED BEANS. *Marlborough Street, London.* Sulphate of copper 2.21 grains per lb. Fine £4 (*B.F.J.*, 1904, 145).

Kensington. Copper equivalent to 1.435 grains of copper sulphate per lb. Fine £2 (*B.F.J.*, 1923, 100).

SPINACH

Two Birmingham samples each contained 0.7 grain of copper per lb. without declaration. A third sample on draining thirty minutes yielded 28 oz. solid and 4 oz. liquid. The solid contained 1.5 grains of copper per lb. It was stated to contain " the smallest quantity possible " of copper. Another sample was a good green, but contained only 0.1 copper.

Willoughby (*B.F.J.*, 1925, 6) has remarked that the chlorophyll is a measure of the vitamin content, and should not be imitated by copper.

PROSECUTION. *London, Bow Street.* Copper 0.027 %, equal to 7.47 grains of sulphate per lb. The Medical Officer of Health said that $\frac{1}{2}$ lb.—or even $\frac{1}{3}$ lb.—would cause a susceptible person to vomit. Fine £5 (*Grocer*, 1907, Dec. 7).

BOTTLED CAPERS

PROSECUTION. *Kingston-on-Thames.* Crystallised copper sulphate 7 grains per lb., which would be injurious to health. Fine 10s. (*P.J.*, 1907, March 23 ; *B.F.J.*, 1907, 68).

CHAPTER XXII

TEA, COFFEE, COCOA, CHOCOLATE

Tea, black, caper. Tea dust. Tea and milk tablets. Coffee, mixtures, French, dandelion. Coffee infusion. Coffee and chicory essence. Chicory. Cocoa, essence. Cocoa and milk powder. Chocolate, chumps, liqueurs, etc. Milk chocolate.

TEA

At one time tea was much adulterated: in 1872 no less than thirty-six out of forty-one Birmingham samples had been adulterated; sand, talc, magnetic iron, China clay, common clay, Prussian blue and foreign leaves were detected. Since 1876 tea has been examined on importation, and a great improvement has resulted (cp. *Analyst*, 1930, **55**, 133). The adulteration of tea samples bought in England and Wales, 1886–1930, divided into three periods, was only 0.2–0.4 %.

In 1874 the Society of Public Analysts suggested that tea after drying should not contain more than 8.0 % of ash, and that at least 3.0 % should be soluble in water (*S.P.A.*, 1878, **2**, 173).

ANALYSES OF "TEA," 1913–30 (300 samples)

Total ash, %	5.1–	5.3–	5.5–	5.7–	5.9–	6.1–	6.3–	6.5–6.8	Total.
Percentage of samples	5	13	29	26	17	6	3	1	100
Ash soluble in water,									
%	2.6–	3.0–	3.2–	3.4–	3.6–	3.8–	4.0–4.2	Total.	
Percentage of samples	2	12	25	40	16	3	2	100	
Ash insoluble in HCl.	0–	0.2–	0.4–	0.6–	0.8–1.8	Total.			
Percentage of samples	35	46	11	5	3	100			

The alkalinity of the soluble ash calculated as % of K_2O was determined on fifty-seven samples, forty of which were between 1.5 % and 1.7 %.

WATER EXTRACT IN "TEA" (116 samples)

Water extract, %		35.6–	38–	40–	42–	44–46.4	Total.
Percentage of samples		3	7	33	38	19	100

All the above results were obtained with undried tea. A few of the samples were inferior, or perhaps somewhat adulterated.

In 1899 there were prosecutions for caper tea yielding up to 13.4 % of ash containing sand and small stones. This tea is said to be manufactured by coating the leaves with gum or starch, and

rolling them up into small balls; mineral matter can easily be enclosed by this process. Analyses of samples have been given by C. Estcourt (*S.P.A.*, 1899, **24**, 30) and J. White (*S.P.A.*, 1899, **24**, 117).

About 1915 grocers were offered a patented preparation, "Roka," for blending with tea to the extent of 20 %. It was said to be compounded from cereals, fruits and nuts and was in the form of black cylinders, 6×1.5 mm., which might easily be mistaken for tea stalks. A sample was found to contain starch; it had a total ash of 2.7 %, and 1.6 % was soluble in water and gave a reddish water extract of 52.8 %. It contained 13.6 % of tannin, which did not support the claim that it reduced the tannin in tea by 20 %. It was sold at 8d. per lb., and its use increased the profit on tea by 2d. a lb. Prosecutions ensued.

Other adulterations or impurities which have been detected are: tea fruit, of which a Birmingham sample contained 8.3 %, exhausted tea, iron filings and lead foil (*Analyst*, 1922, **47**, 478), also excess of tea stalks (Besson, *Analyst*, 1911, **36**, 454; Deuss, *Analyst*, 1916, **41**, 78).

In 1929-30 several samples of tea bought in Birmingham had labels stating "no crude tannin" or "remarkably free from the objectionable tannic acid," or similar statement, while 12.5-14.9 % was present. As eighteen samples, for which no such claims were made, contained 8.6-13.9 % of tannin, with an average of 11.4 %, these labels were condemned as "false." The packers were cautioned, and undertook to alter the labels. An article in the *Lancet* (1911, Jan. 7, Dec. 2; also *Grocer*, 1911, Jan. 11, Dec. 16) emphasised the importance of the tannin being in the form of caffeine tannate, and that the caffeine and the tannin should be in the proportion of 1 to 3. H. L. Smith has given the composition of infusions prepared in different ways. He found that hardness in water decreased the extraction of tannin, but did not effect the extraction of caffeine (*P.J.*, 1913, June 28). There was a considerable amount of correspondence on "Tannin in Tea" in the *Grocer* during August to October, 1930. Anderson has discussed the factors which constitute value in tea (*J.S.C.I.*, 1909, 285).

In 1923 the Customs analysts detected arsenic in green tea, which was probably due to the use of ferrocyanide of potassium for facing it, and a small quantity of the tea was recalled from distribution (*Grocer*, 1923, Feb. 17).

Carter Bell has published ash determinations of fifty-eight samples of tea (*S.P.A.*, 1882, **7**, 7), and Tatlock and Thomson have analysed twenty-two samples of tea and the infusions made from them (*S.P.A.*, 1910, **35**, 103).

ANALYSIS. The methods for determination of *soluble ash* and its *alkalinity* have previously been given (p. 71). The use of a

magnet for the detection of iron or magnetic oxide of iron is sometimes useful. Determine *water extract* by boiling 1 gm. of finely powdered tea with 400 ml. of water under a condenser for one hour, filtering, and weighing the extract from 100 ml. This proportion is better than using 1 w/v, which precipitates, more or less, on cooling during filtration. The insoluble leaf may be determined by drying the residue; four Birmingham samples gave 52–59 %. This determination is useful in the analysis of tea tablets. H. L. Smith has studied the cinchonine method for determination of *tannin* (*S.P.A.*, 1913, **38**, 312) and Tatlock and Thomson (*opus cit.*) have described the quinine method for it, and given methods for other determinations.

PROSECUTIONS FOR TEA. *Stockton.* Mineral matter more than genuine 9 %, principally magnetic oxide of iron and sand. Fine £1 (*Analyst*, 1878, **3**, 296).

Nottingham. Coloured starch known as “Roka” 20 %. Fine £5 (*Grocer*, 1915, Nov. 13; *B.F.J.*, 1915, 218).

Kendal. Being dealers in tea mixed with an ingredient known as “Roka,” described as a patented substance compounded of cereals, fruits and nuts, contrary to the Act of 1724. Two fines of £25 (*Grocer*, 1918, Nov. 2; *B.F.J.*, 1918, 126).

West Ham. Manufacturing 50 lb. of used tea leaves in imitation of tea, and being in possession of 35,550 lb. of tea leaves which had been manufactured in imitation of tea, contrary to the Adulteration of Tea Act, 1776. Fine £250 (*Grocer*, 1917, Dec. 29; 1918, Jan. 26; *B.F.J.*, 1918, 16, 29).

Southend. Iron filings 2·7, 6·2 and 3·8 grains per oz., respectively. Its presence was attributed to “sweepings” from a warehouse. Total fines £30 (*Grocer*, 1926, Oct. 9; *Analyst*, 1926, **51**, 573).

Salford. Dust 12 %, which should not exceed 2 %. The dust included 6 % of chalk, sand and silicious matter; 8 grains per lb. of iron filings were also present. Fine £5 (*Grocer*, 1927, Jan. 29).

PROSECUTION FOR BLACK TEA. *Birmingham.* Plumbago and “lie” tea. Fine £2. The ash was 8·6 %; 3·0 % of magnetic iron was present. (1874 Report).

PROSECUTION FOR CHINA TEA. *Poole.* Exhausted tea leaves 20 %, extraneous mineral matter 2 %. It was said to be a mixture of two teas, one of which might be mistaken for used tea. Another analyst described it as dirty tea, but without exhausted leaves. Case dismissed (*Grocer*, 1918, Feb. 23; *B.F.J.*, 1918, 45).

PROSECUTIONS FOR CAPER TEA. *Alfreton.* Mineral matter 8 %, of which 6 % was small stones wrapped up in leaves. Fine £5 (*Analyst*, 1877, **1**, 33).

Chesterfield. Mineral matter, sand and small stones 3·5 %. The Public Analyst stated that sixty-five samples of caper tea contained less than 2 % of sand and stones, and only six as much

as 3 %. The magistrates declined to convict the defendant on so small a percentage of extraneous matter of a nature peculiarly incident to the growth of caper tea on wet sandy soils. On appeal, *Shortt v. Robinson* (1899), it was held that the magistrates acted on some knowledge of their own, as they were entitled to do, and the appeal was dismissed (*Analyst*, 1899, **24**, 120 ; *B.F.J.*, 1899, 52).

Derby. Ferruginous sand and small stones 6.26 %, while 3 % was a liberal allowance. Total ash 13.47 % instead of a limit of 8 %. Another analyst found 6.16 % and 13.4 %, respectively. The Government analysts reported 6.4 % of sand and silica, and that the extraneous matter had been unavoidably mixed during collection or preparation. One of them stated that the Customs authorities passed caper tea containing 13–14 % of total ash. (This statement was subsequently shown to be incorrect (*Analyst*, 1899, **24**, 333).) Case dismissed and 2 guineas costs allowed to defendant (*B.F.J.*, 1899, 126, 148, 170 ; *Analyst*, *opus cit.*).

PROSECUTION FOR TEA DUST. *London, Old Street*. Sand or fine grit 6 %. The Public Analyst stated that the total ash was 13.7 %, of which 7.15 % was insoluble in acid. The magistrate considered the offence trifling and bound the defendant over under the Probation Act (*Grocer*, April 16, 1910).

PROSECUTION FOR TEA AND MILK TABLETS. *Bishop Auckland*. Carbonate of lime 5 %, starch 15 %. The defendants said they used French chalk and starch, not lime. Paid costs (*Grocer*, 1918, Dec. 28 ; *B.F.J.*, 1919, 15).

COFFEE. CHICORY

For practical purposes, adulteration of coffee may be described as solely due to the addition of chicory, but analyses of artificial berries have been given by Maljean (*Analyst*, 1897, **22**, 17) and Cribb (*S.P.A.*, 1902, **27**, 114) ; the latter also detected starch in ground coffee. In 1908, eighty-four sacks of coffee beans were seized in the United States as injurious to health ; their inferiority had been concealed by coating with lead chromate. The same adulterant was detected in a number of samples bought in Malta (*B.F.J.*, 1899, 46). Chicory has been adulterated with mineral matter or sand, and in one instance coffee was substituted.

The following analyses refer to Birmingham samples :—

MOISTURE IN COFFEE (1,235 samples) AND CHICORY

MOISTURE, % .	1-2-	3-	4-	5-	6-	7-	8-	9-	10	Total.
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PERCENTAGE OF SAMPLES

Coffee	2	16	30	25	17	5	4	1	0	100
Chicory	0	2	7	12	19	16	13	7	24	100

As coffee and chicory have been roasted, the amount of moisture should be small, say under 7 %, but moisture may be absorbed if they are kept in drawers (see Hodgson, *S.P.A.*, 1913, **38**, 454). These articles should be kept in closed tins so that purchasers are not defrauded by receiving them containing an excessive amount of water and being deficient in aroma. Beans that have been treated with a solution of borax may yield a roasted coffee containing over 10 % of moisture (Dodd, *S.P.A.*, 1927, **52**, 463). Pritzker and Jungkunz (*Analyst*, 1926, **51**, 355) suggest a limit of 7-8 % for coffee. If action be contemplated for excess of water, care should be taken over the sampling to prevent increase of water.

TOTAL ASH IN COFFEE (Sixty-three samples) AND CHICORY
(Seventy-six samples)

ASH, %	3-6	4-0	4-5	5-0	6-0	7-0	8-0	9-0-10-6	16-9-18-0	Total
PERCENTAGE OF SAMPLES										
Coffee	18	68	13	1	0	0	0	0	0	100
Chicory, 1917-21	0	8	10	25	13	13	15	8	8	100
1924-31	0	8	22	43	16	3	5	3	0	100

ASH INSOLUBLE IN ACID IN COFFEE (Thirty-five samples)

Insoluble ash, %	0-	0-1-	0-2-	0-3-0-4	Total
Percentage of samples	43	20	31	6	100

ASH INSOLUBLE IN ACID (SAND) IN CHICORY (Eighty-one samples)

INSOLUBLE ASH, %	0-	0-5-	1-0-	1-5-	2-0-	3-0-	4-0-5-5	11-0-11-8	Total
PERCENTAGE OF SAMPLES									
1917-21	0	31	5	13	20	8	15	8	100
Other years	26	17	12	21	17	5	2	0	100

Coffee, being prepared from a bean, has no natural sandy matter ; samples of coffee berries yielded 0-02-0-06 % of ash insoluble in acid. Chicory, on the other hand, is obtained from a root, and the large amounts of total ash and sand, given above, which were found in the war-time chicories (1917-21) were due to the roots, either from carelessness or intention, being inefficiently washed. The sale of sand at 1s. 8d. or 2s. a pound was a fraud on the public. Ten of the twenty-eight Birmingham samples examined in 1917 contained more than the liberal allowance of 8 % of total ash. Other years were much better. Of the samples examined in England and Wales during 1920-6, 7 % were reported adulterated.

ASH SOLUBLE IN WATER IN COFFEE AND CHICORY
(Ninety-one Samples)

SOLUBLE ASH, %	1-6-	2-0-	2-5-	3-0-	3-5-3-7	Total
PERCENTAGE OF SAMPLES						
Coffee	0	0	11	50	39	100
Chicory, 1917-21	3	36	46	12	3	100
„ other years	20	25	30	23	2	100

The alkalinity of the soluble ash, N. v/w, was 39-42 for coffee, and 15-18 for chicory, determined on the dried substances.

SOLUBLE EXTRACT IN DRIED COFFEE (2,624 samples)

SOLUBLE EXTRACT, %.	20-	23-	24-	25-	26-	27-	28-30.3	Total.
PERCENTAGE OF SAMPLES								
1896-1917 . . .	2	12	25	33	19	8	1	100
1918-29 . . .	4	17	35	29	12	3	0	100

SOLUBLE EXTRACT IN DRIED CHICORY, 1924-31

(Thirty-seven samples)

Soluble extract, % . . .	52-	70-	72-	74-	76-	78-80	Total.
Percentage of samples . . .	8	5	19	14	46	8	100

The two lowest samples of chicory were high in total ash, containing 8.6 % and 10.6 %, respectively. An over-roasted sample of nibs yielded only 67.2 % of soluble extract. Dyer has given analyses of a number of samples of chicory which show that the percentage of insoluble matter is mainly a function of the mode and duration of roasting (*S.P.A.*, 1898, **23**, 226).

Dyer also suggested that the high soluble extract (32 %) in some samples of coffee may be due to the addition of sugar during roasting. Pritzker and Jungkunz (*Analyst*, 1926, **51**, 355) approve of a minimum limit of 20 % for coffee.

Tatlock and Thomson have given a paper on the analysis and composition of coffee and chicory (*J.S.C.I.*, 1910, 138). Clayton has given analyses of chicory and dandelion (*S.P.A.*, 1895, **20**, 12) and of roasted beetroot (*S.P.A.*, 1904, **29**, 279). He found 0.014-0.039 % of caffeine in coffee-palace infusions of coffee (*S.P.A.*, 1897, **22**, 172). Pritzker and Jungkunz (*opus cit.*) have given particulars of caffeine-free coffee, and also analyses of chicory and dandelion (*Analyst*, 1921, **46**, 289).

Coffee Mixtures. As early as 1853, an Order for the protection of the Excise required coffee mixtures to be marked as such, with letters of a definite size. It seems reasonable to expect that a mixture marked "Coffee and Chicory" should contain at least half of the first-named constituent. Of twenty-one Birmingham samples thus labelled, fourteen contained 36-50 % of *chicory*, two had a little over 50 %, and one 70 %. Of the thirteen samples labelled "Chicory and Coffee," seven contained less than 50 % of chicory, and one as much as 81 %. Eight of the samples contained 45-60 %. It has been stated in prosecutions that it is difficult to make a uniform mixture of the two articles. It is therefore important that samples should be thoroughly mixed by the purchaser before division for analysis. On one occasion the three parts of a Birmingham sample were each analysed. One contained about 27 % of chicory, the second 5 %, and the third very little.

At one time attempts were made by vendors of coffee in tins to annex the term "French Coffee" for an article containing large proportions of chicory, *without any label* to indicate the article was a mixture. For example, a tin labelled "Fine French Coffee" contained 97 % of chicory. Mixtures containing a considerable proportion of chicory have been sold as "Dandelion Coffee."

In coffee mixtures it is important to consider if, at the price charged, the amount of the cheaper constituent is sufficient to make it a fraudulent addition to increase the bulk. For the guidance of a purchaser it would be useful if there was a definite statement on the label of the proportion of coffee present. If the proportion present did not agree with that stated on the label, a prosecution might be instituted for false label under sect. 30 of the 1928 Act, but it is necessary to prove that the action was "wilful."

Adulteration of Coffee with Chicory. There has been a great fall in Birmingham of this form of adulteration. In 1882-9 no less than 54 % of the samples of coffee were adulterated. In 1895-1900 the proportion had fallen to 10 %; £96 was paid in fines. During 1905-30 only 1.6 % of the samples were adulterated, in some cases due to accident. In the earlier period the average percentage of chicory was 53 %, against 26 % in the later.

In only two of the years, 1877-1892, was the percentage of adulteration of coffee in England and Wales below 15 %. From 1893 to 1913 there was a gradual fall from 11.6 % to 3.7 % of adulteration. During 1919-30 the average was 0.9 %. Of the "coffee mixtures" 9.4 % were adulterated during 1905-13.

It is obvious from the variations in the composition of samples of coffee and of chicory previously given, that the determination of the proportion of the two in a mixture is very difficult. In 1882 the Birkenhead Corporation arranged for three mixtures of coffee and chicory to be made and each to be examined by seven analysts. The first contained 10 % of chicory, and the reports indicated from 0 to 16 %. The second sample contained 30 %, and from 25-35 % was reported. The third had 37½ %, but was reported to have from 34-50 % (*Analyst*, 1882, 7, 76). Part of the differences may possibly have been due to imperfect mixing, but it is obviously unwise for an analyst to be too rigid in certifying such adulteration.

The determination of caffeine—the characteristic constituent of coffee—may be useful, particularly for infusions and essences. The determination is, however, tedious, and, according to Lendrich and Nottbohm, the amount may vary from about 1 % to 3 % (*Analyst*, 1909, 34, 214, 484). As a fair average, 1.2 % has been taken.

Chicory infusions have about three times the tinctorial power of those made with coffee (Allen, *Chem. News*, xxix., 40). Pritzker and Jungkunz (*Analyst*, 1921, 46, 289) recommend N/20 iodine as

a suitable standard, it having about the colour of 5 % decoction of chicory. The colour yielded by chicory depends on the amount of roasting, and caramel may have been added.

The determination of fat and sugar in a mixture may be useful, but as lard is added to chicory during roasting, and as sugar may be added to chicory (Clayton, *opus cit.*) or to coffee, calculations from these constituents may be deceptive.

The amount of soluble extract in the dried mixture is probably the most reliable figure, and the following formula (cp. p. 107) is suggested :—

$$\text{Chicory, \%} = \frac{(\text{Soluble extract} - 25)100}{76 - 25}.$$

$$\text{Or, } 1.96 (\text{soluble extract} - 25).$$

It must be remembered that the formula depends on the assumption that no *third* substance is present. The perhaps accidental presence of 5 % of insoluble substance, as starch, in a mixture of equal parts of chicory and coffee would cause the chicory to be underestimated by 2.5 %. The presence of 5 % of sugar would cause the chicory to be overestimated by 7.5 %. As coffee grounds were found to contain 5.5 % of soluble extract, a mixture of 40 % of such grounds with 15 % of chicory, and 45 % of genuine coffee, would give a normal figure for soluble extract, though the mixture would be deficient in taste.

The determination of the sp. gr. of a 10 % infusion is a less accurate duplication of the soluble extract. The ash soluble in water of coffee is usually higher than that of chicory, but as shown above, there is overlapping. Chicory contains more chlorine (0.15 %) than coffee (0.03 %) but the figures are small. Sand in coffee probably indicates chicory, but its presence may be accidental. For a figure quite independent of the soluble extract the writer suggests the determination of the alkalinity of the soluble ash, and the following formula, but considerable care is necessary in the determination :—

$$\text{Chicory, \%} = \frac{(41 - \text{alkalinity of soluble ash, N.v/w})100}{41 - 16}.$$

As in the previous formula, the absence of a *third* substance is assumed, but as neither starch nor sugar will yield an appreciable amount of alkalinity, the apparent adulteration will be increased by the addition of either of them. As coffee grounds were found to give a figure of only 3.3 N.v/w, the above-mentioned mixture giving a normal figure for soluble extract would give an alkalinity corresponding to 67 % of adulteration. Comparison of the amounts of adulteration indicated by the soluble extract and the alkalinity should indicate any abnormality in the composition of the mixture.

As the ratio between soluble extract and alkalinity is not constant either in coffee or chicory, too close agreement in the two calculations must not be expected.

ANALYSIS. *Soluble Extract.* To extract *completely* the soluble matter of chicory and coffee many treatments are necessary, but the following method leaves little in the residue. Add 150 ml. of boiling water to 5 gm. in porcelain dish, heat to boiling and keep nearly boiling for fifteen minutes. Decant through coffee strainer into 250 ml. flask. Wash residue into dish with 50 ml. boiling water, nearly boil for five minutes as before, strain, etc. Repeat extraction twice more, which should give nearly 250 ml. of liquid. When cold dilute to 250 ml., filter, evaporate 50 ml. in flat-bottom metal dish, and dry in water oven three to four hours. Calculate to dry coffee, or use dried coffee for the determination.

Alkalinity of Soluble Ash. Ignite 5 gm. over lowest possible luminous flame and ignite the vapour. Chicory requires no further heat, but otherwise continue the heat for two minutes after the flame has ceased. Add 50 ml. of water to the dish, put on water bath for about five minutes, then filter. Ignite the filter paper and residue over luminous flame turned low till the carbon is burned off. Wash the residue from the platinum dish into a porcelain dish with 100 ml. of water and keep just boiling for five minutes. Filter into the flask containing the previous filtrate, and wash residue twice with about 10 ml. of water. Titrate with N/10 HCl, using methyl red as indicator. Doubling the figure will give N.v/w. Calculate to dry coffee.

Examination for Chicory. In his early days the writer trusted to the "floating test," which consists in putting some of the sample on the surface of water, and noticing if part sinks and colours the water. He had, however, the unpleasant experience of being told by an inspector that a sample passed as genuine had been stated by the vendor to contain chicory! When only a few per cents. of chicory are present, soft white particles are evident on the surface of the residue of the sample which has been extracted for soluble extract. On microscopical examination of these particles the majority of the vessels are seen to be reticulated, the width being about 20–100 μ , but usually about 50 μ . The larger vessels have transverse walls, 60–200 μ apart, but the smaller do not show them. Spiral vessels may sometimes be seen 10–13 μ wide. The ends of the vessels occasionally appear pointed where they join. The parchment membrane called "flights" is quite different in appearance.

Dandelion Root. The vessels seen in two fresh and two dried samples were chiefly spiral, the width ranging from 20–60 μ , but usually 30–40 μ . There are no visible transverse walls, or pointed ends of vessels. It has been stated that *roasted* dandelion root

cannot be distinguished from roasted chicory root, but the writer has had no opportunity to check the statement.

Detection of Starch. Boil with water, strain, add H_2SO_4 and destroy the colour with KMnO_4 solution, then test with iodine (Allen, *S.P.A.*, 1880, 5, 3).

APPEAL CASES ON COFFEE. *Liddiard v. Reece* (1880). The vendor asked for "coffee" and was supplied with an article containing 60 % of chicory, and labelled "This is sold as a mixture of chicory and coffee." The vendor had been fined £5 for a sale to the prejudice of the purchaser, as (1) the article was not of the nature, etc., of "coffee." (2) Coffee price being charged, the chicory was fraudulently added to increase the bulk. (3) The label was not pointed out before the sale was completed. The conviction was confirmed, the High Court holding that notification on the label did not protect the seller if there had been fraudulent addition to increase the bulk (*Analyst*, 1880, 5, 10). *Horder v. Meddings* (1880) was a similar case (*Analyst*, 1881, 6, 214).

Higgins v. Hall (1886). The vendor said she did not sell "coffee," but offered a mixture "coffee and chicory" which contained 30 % of coffee. The High Court quashed the conviction, as the vendor was entitled to sell a mixture.

A vendor was fined £3 for selling a mixture containing 60 % of chicory as "coffee" when labelled "This is sold as a mixture of chicory and coffee." The conviction was confirmed on appeal to London Quarter Sessions, *Jones v. Vestry of Bermondsey*. In the judgment it was stated that coffee should be described as "food," as it was not a "drink" till water was mixed with it. The mixture was considered to be fraudulent, as the label did not suggest so large a proportion of the cheaper article, and that the profit was 25 %, while if the articles had been sold separately, it would have been 17 % (*Analyst*, 1892, 17, 157).

Hewson v. Gamble (1892) decided that a vendor who snatched a purchased parcel of coffee did not commit larceny.

PROSECUTIONS FOR COFFEE. *Gainsborough.* Sugar 17 % besides chicory. Fine 10s. (*Analyst*, 1878, 3, 368).

Wolverhampton. Practically all chicory. Case dismissed as no percentage given (*Analyst*, 1880, 5, 189).

Birmingham. Chicory, 90 %. Fine £1 (1895 Report).

North London. Chicory 85 %; another public analyst found 60 %, and the Government analysts 52 %. The defendants admitted that in the "shilling coffee" there was 50 % of chicory, and pleaded that coffee and chicory were difficult to mix. The packet was labelled "Coffee and Chicory." The defendant was fined £3, but the decision was reversed on appeal to Sessions, as the Bench had not found that there had been absolute fraud (*F. & S.*, 1898, Feb. 5, 26; April 23).

Birmingham. Chicory 14 %. The shop assistant had weighed $\frac{1}{4}$ lb. coffee berries, and ground them in a mill previously used for grinding chicory. The magistrates thought there had been carelessness, but not fraud. Fine £1 (*B.F.J.*, 1899, 55).

Cookstown. Entirely chicory. The Government analysts found 1 % of coffee. It was sold in a tin. Fine £4 (*Grocer*, 1905, Nov. 18).

Manchester. Foreign matter, probably chicory 48 %. Fine 10s. (*Grocer*, 1909, Feb. 27).

Colne. Roasted rice 5 %. Fine £1 (*B.F.J.*, 1910, 71).

West London. Foreign vegetable matter, not nuts, nor chicory. Fine £1 (*Grocer*, 1913, Feb. 15).

London, Marylebone. Chicory 60 %. The tin was labelled "French Coffee, a blend of the finest chicory and coffee." As the price charged was almost exactly that of the ingredients, the magistrate found that there had been no fraudulent addition to increase the bulk, and dismissed the case, allowing the defendant 23s. costs (*Grocer*, 1914, Jan. 10; *B.F.J.*, 1914, 12).

London, Bow Street. False trade description "Practically caffeine-free, 100 oz. containing 1/100 oz. of caffeine." The Public Analyst said that 1.02 % was present, being rather below the average (1.2 %). Evidence was given for the defence that the caffeine found was not free caffeine but derived from caffeine salts. Case dismissed (*Analyst*, 1929, 54, 469; *B.F.J.*, 1929, 65, 76).

London, Old Street. Chicory 65 %. The defendant said that the inspector pointed to a tin on the stall and asked for " $\frac{1}{4}$ lb. of that." The inspector said he asked for coffee. Fine £5 (*Grocer*, 1929, Nov. 18; *B.F.J.*, 1930, 4).

PROSECUTIONS FOR COFFEE MIXTURES. *West London.* Starch 10 %. The defendant believed the article was a mixture of chicory and coffee, neither of which contains starch. The definition of "coffee mixture" in the Inland Revenue Act, 1882, was quoted, but the magistrate ruled that it did not include starch, and ordered the defendant to pay £5 10s. (*B.F.J.*, 1901, 240, 299).

Marlborough Street. Chicory 68 %, being 23 % more than was notified on the label. The Government analysts found 66 %. The magistrate dismissed the case, as the purchaser did not ask for particular proportions, and allowed the defendant 2 guineas costs (*Grocer*, 1906, June 23; *B.F.J.*, 1906, 137). (*Note* : A prosecution for false label might have been successful.)

Nottingham. Chicory 85 %. The magistrates held that the purchaser was not prejudiced; he might have enquired, but did not, what were the proportions. The price was not excessive, and the addition did not fraudulently increase the bulk. Case dismissed (*Grocer*, 1909, May 1, 22; *B.F.J.*, 1909, 90).

APPEAL CASE ON FRENCH COFFEE. *Caistor.* Chicory 60 %. As he was being paid the vendor said, "This is sold as a mixture of

coffee and chicory," and pointed out the label on the tin. The manufacturer said he had sold "French coffee" for many years, and that after trials he had found half-and-half suited the public taste, and that was the composition of the sample. Other qualities had 90 % of chicory. The magistrates fined the defendant £5 as, the proportion of chicory not having been stated, it must have been added fraudulently to increase the bulk. The High Court, *Otter v. Edgley* (1893), held there was no evidence to support a conviction (*F. & S.*, 1893, Jan. 14).

PROSECUTION FOR DANDELION COFFEE. *Swansea*. Chicory 75 %, coffee 25 % and no dandelion. Evidence was given of the difficulty of distinguishing the roasted roots of chicory and dandelion. Dismissed (*F. & S.*, 1892, Aug. 27). In a similar case the maker gave evidence that he used dandelion root (*F. & S.*, 1893, April 22).

PROSECUTION FOR COFFEE INFUSION. *Lambeth*. Made from a mixture of 70 % of chicory and 30 % of coffee, and sold at a coffee-shop. Fine 10s. (*F. & S.*, 1896, 189).

PROSECUTION FOR CHICORY. *Birmingham*. Vegetable matter foreign to chicory 100 %. The sample was a coarse-grained, slightly roasted coffee, of inferior quality, which had been left over from war-time. Fine 10s. (*Grocer*, 1924, July 12; *B.F.J.*, 1924, 75).

COFFEE AND CHICORY ESSENCE

The examination of six Birmingham samples in 1929 showed that the article is of very variable composition. The total solids varied 57.9-68.5 %, of which 28.5-52.0 % was sucrose, and 4.8-23.8 % invert sugar, and ash 0.8-1.5 %. The caffeine in three samples varied 0.18-0.32 %. Neither borates nor benzoates were detected.

A solid proprietary article proved to be a dried aqueous extract of coffee, and contained 5.76 % of caffeine. The label on the tin claimed that the contents (1 oz.) were equivalent to more than $\frac{1}{2}$ lb. of ordinary coffee. As only about one-quarter of coffee is soluble in water, the label was condemned as a false one, and the vendors undertook to alter the description.

Analyses of coffee extracts have been given by Moor and Priest (*S.P.A.*, 1899, **24**, 281), Tatlock and Thomson (*J.S.C.I.*, 1910, 138), and Ponce and Palma (*Analyst*, 1921, **46**, 136).

PROSECUTION FOR A BOTTLE OF COFFEE (COFFEE ESSENCE). *Hamilton*. Prepared from 1 part of coffee and $2\frac{1}{2}$ parts of chicory, with the addition of sugar. The bottle was labelled "Finest Essence of COFFEE with Chicory." Evidence was given that purchasers of a "bottle of coffee" expected to be supplied with a mixed article containing a large proportion of sugar, to

prevent it fermenting. The sheriff considered the label gave due notice, and that at the price at which the article was sold, it was difficult to charge the vendor with fraud to any extent. Charge not proven (*Grocer*, 1915, June 19, 26; *B.F.J.*, 1915, 139).

COCOA

Strictly speaking, "cocoa" is cocoa nibs—cocoa beans roasted and deprived of shell. As about half of the nibs is fat, in the old days they were boiled with water and the fat skimmed off, or the beverage would be too rich for the stomach. The "prepared," or "pearl" cocoas, which were very popular at one time, consisted of about equal parts of cocoa, sugar, and arrowroot or sago starch. The additions made the liquid thick and hindered the rising of the cocoa fat. Sometimes part of the fat was improperly removed from the cocoa. Such articles have been described as "chocolate powder," but the better name is "cocoa mixture." They have been largely superseded by "cocoa essence," composed of cocoa nibs of which part of the fat has been removed by pressure.

During war-time cocoa shell was sold under fancy names at excessive prices; the following table gives the average of a few Birmingham analyses and of cocoa nibs:—

AVERAGE PERCENTAGE COMPOSITION OF COCOA NIBS AND SHELL

	Cocoa Nibs.	Cocoa Shell.
Moisture	3.3	9.4
Fat	51.9	2.5
Nitrogen	2.1	2.6
Total ash	2.6	8.4
Ash soluble in water	—	5.1
Ash insoluble in HCl	—	1.1
Phosphate, P_2O_5	0.9	0.6
Cold water extract	10.4	21.4
Organic	7.9	16.6
Ash	2.5	4.8
Alkalinity of soluble ash, N.v/w	21	49

A. W. Knapp informs me that roasted cocoa nibs now usually contain 54–55 % of fat. Booth, Cribb and Richards have given analyses of a number of samples of cocoa nib and shell (*S.P.A.*, 1909, **34**, 143); Knapp has given a paper on the separation and uses of cacao shell (*J.S.C.I.*, 1918, July 31); Baker and Hulton have analysed a number of "cocoa teas" (*S.P.A.*, 1918, **43**, 189). For an analysis of an infusion made from shells, see Cribb (*S.P.A.*, 1918, **43**, 203).

In 1890–9 the sale of cocoa mixtures as "cocoa" was common, the percentage of adulteration in England and Wales varying from 31–76 %. The average of 1904–13 was 6.7 %, and during 1919–30 it fell to 0.6 %. For articles sold as "chocolate powder" or "cocoa mixture," the percentage of adulteration during 1905–12

was 1·9 %, and during 1920-30 only one of the 143 samples examined was condemned.

Cocoa Essence. The following tables give the composition of Birmingham samples :—

MOISTURE IN COCOA ESSENCE, 1921-9 (Eighty-four samples)

Moisture, %	3·5-	4·5-	5·5-	6·5-	7·5-8·0	Total.
Percentage of samples	7	20	41	20	12	100

FAT IN COCOA ESSENCE, 1918-30 (134 samples)

Fat, %	17·7-	20-	22-	24-	26-	28-	30-32·9	Total.
Percentage of samples	8	21	24	14	11	16	6	100

TOTAL ASH IN COCOA ESSENCE, 1918-30 (141 samples)

Total ash, %	4·5-	5·5-	6·0-	7·0-	7·5-8·0	Total.
Percentage of samples	14	10	29	38	9	100

ASH SOLUBLE IN WATER IN COCOA ESSENCE, 1922-30
(Eighty-seven samples)

Ash, soluble in water, %	4·2-	4·6-	5·0-	5·4-	5·8-6·4	Total.
Percentage of samples	14	10	29	38	9	100

ASH INSOLUBLE IN HCl IN COCOA ESSENCE, 1919-30 (141 samples)

Ash insoluble in HCl, %	0·02-	0·1-	0·2-	0·3-	0·4-0·7	Total.
Percentage of samples	23	54	13	7	3	100

Of the eighteen samples examined sixteen contained 17-20 % of cold water extract.

FIBRE IN DRY FAT-FREE COCOA ESSENCE, 1921-7
(Forty-eight samples)

Fibre, %	5·5-	5·8-	6·0-	6·2-	6·4-	6·6-6·7	7·0-7·7	Total.
Percentage of samples	17	12	17	14	19	15	6	100

The samples containing 7·0-7·7 % of fibre were condemned as containing an excess of shell. If the amount of ash insoluble in HCl is under 0·1 %, excess of shell is probably absent. A. W. Knapp informs me that the proportion of fibre has risen slightly and is now about 6 %.

Baker and Hulton have examined some methods for the determination of shells (*S.P.A.*, 1918, **43**, 197). Knapp and McLellan have examined eleven methods and criticised them in an exhaustive paper on the estimation of cocoa shell (*S.P.A.*, 1919, **44**, 2).

The Cocoa Powder Order, 1918 (*Analyst*, 1918, **43**, 248), made by the Food Controller, made a distinction between cocoa powder containing 5 % of shell, and that containing 2 %, but Knapp and McLellan (*opus cit.*) showed that no analytical method would distinguish them.

For particulars of cocoas which have been treated with alkali

to make them more soluble, see Rocques (*Analyst*, 1917, **42**, 173, 391; 1918, **43**, 217). Arsenic is a possible impurity in the alkali used, and the Report of the Ministry of Health for 1922 states that twenty-six samples of cocoa out of 1,851 contained more than 1/100 grain of arsenic per lb.

Calculation of the Composition of Mixtures. For the purpose of calculation, it may be taken that cocoa nib contains 2.1 % of nitrogen, and 0.9 % of phosphate (P_2O_5). The total ash is about 2.6 %, and the ash of the cold water extract about 2.5 %, but the addition of iron oxide to diminish the pale colour, or of alkali, may render these figures useless.

The use of the above constants assumes that no fat has been removed from the cocoa, a supposition which may be incorrect. The following formula avoids that error :—

(A) Percentage of cocoa = $N \% \times 20 + \text{Fat \%} + \text{Moisture \%}$.

The percentage of sugar may be obtained by polarisation, or by the following formula :—

(B) Percentage of sugar =
$$\frac{(\text{Organic cold water extract \%} - 8) 100}{100 - 8}$$
.

The determination of fibre may be necessary to prove the absence of shell. Analyses of a test mixture made in the laboratory and of two bought samples illustrate the method :—

ANALYSES OF COCOA MIXTURES

Percentages.	Test Mixture.	Sample I.	Sample II.
Moisture	3.4	6.7	7.8
Fat	20.9	6.9	3.8
Nitrogen	0.83	0.92	0.46
Total ash	1.12	1.09	2.60 (red)
Phosphate (P_2O_5)	0.41	0.25	0.21
Cold water extract, total	45.4	44.6	44.0
Organic	44.3	43.6	41.8
Ash	1.1	1.0	2.2

PERCENTAGE OF COCOA :

By formula (A)	43	32	21
From phosphate : ($P_2O_5/0.009$)	46	23	23
From nitrogen ($N/0.021$)	40	44	22
Actual	40	—	—

PERCENTAGE OF SUGAR :

By formula (B)	40	39	39
By polarisation :			
Sucrose	39	35	31
Dextrose	1	7	2
— 40	— 40	— 42	— 33
Actual	40	—	—

PERCENTAGE OF STARCH :

By difference	17	28	42
Actual	20	—	—

In sample I. the high figure calculated for cocoa from the nitrogen, and the low one from the phosphates, suggest the presence of cocoa shell in the sample.

ANALYSIS. *Fat.* Weigh 1–2 gm. according to the nature of the sample. Grind up with sand in a mortar, transfer mixture to flask, and wash out mortar with 50 ml. petroleum spirit. Shake during day and filter next morning. Wash flask and filter thrice with 10 ml. of the solvent. Evaporate to dryness, and dry fat about two hours. Repeat washing and evaporate filtrate in another dish. If the extracted fat exceeds a few mgm., repeat the washing. If the fat be determined in a Soxhlet, prolonged extraction (twelve hours or more) of the fine powder is necessary.

Cold Water Extract. Rub up 5 gm. in a mortar with 250 ml. of water, transfer to flask, shake occasionally during a day, and filter next morning. Evaporate 50 ml. to dryness, and dry about two hours for total extract, and take 100 ml. for ash. The alkalinity of the ash may be determined, using methyl orange as indicator.

Sugars. Rub 15 gm. in mortar with 5 ml. methylated spirit, add 35 ml. water, and filter after standing fifteen minutes. Wash till filtrate is about 90 ml. Add basic lead acetate solution, alum solution, alumina cream, and water to make 100 ml. Filter and polarise, making acid if necessary with acetic acid. Invert and polarise again.

Nitrogen. Determine on 5 gm. of cocoa mixture, 3 gm. of nibs, or 2 gm. of cocoa essence. For the determination of starch by taká-diastase, see Revis and Burnett (*S.P.A.*, 1915, **40**, 429); and for theobromine, papers by Radford and Brewer (*S.P.A.*, 1917, **42**, 274), and Wadsworth (*S.P.A.*, 1920, **45**, 133; 1921, **46**, 32; 1922, **47**, 152). The methods for the analysis of chocolate given by Booth, Cribb and Richards (*S.P.A.*, 1909, **34**, 140) are mostly applicable to cocoa.

PROSECUTIONS FOR COCOA. *Clerkenwell.* Sugar 40 %, starch 15 %. Case dismissed as it was sold as “chocolate powder” (*F. & S.*, 1893, March 4).

Ystrad. Added starch and sugar 70 %. It was “pearl” cocoa, and was labelled “Cocoa, combined with other ingredients.” It was handed to the purchaser in an opaque wrapper. The manufacturer stated that sago was present, and not starch. Fine ls. On appeal to Quarter Sessions, the Court failed to agree. On appeal to the High Court, *Jones v. Jones* (1894), the conviction was quashed as there was sufficient notice of mixture, and no evidence of fraudulent increase of bulk (*F. & S.*, 1893, June 10, July 8; 1894, March 10). *R. v. Field* (1895) was a similar appeal case.

Birmingham. Sugar 40 %, arrowroot 30 %; sugar 40 %, sago starch 15 %. Each vendor, and two others who sold similar articles, were fined £2 (*F. & S.*, 1897, July 17).

Shrewsbury. Sugar 35 %, starch 20 %, oxide of iron or raddle 0.56 %. The article was not labelled. Fine £5 (*F. & S.*, 1898, April 9).

London, South-Western. Cocoa husk 20 %, sugar 40 %, farina 40 %. Cocoa was absent. It was labelled "Chocolate powder. Fine cocoa combined with other ingredients." Fine 10s. (*Grocer*, 1907, May 4 ; *B.F.J.*, 1907, 84).

West London. Powdered cocoa shell 60 %. The Government analysts subsequently reported the presence of not less than 65 % of cocoa husk coloured by aniline dye. Evidence was given that the shell was removed from the roasted beans before making into commercial cocoa. After a long hearing the vendor was fined £2 (*Grocer*, 1910, July 30 ; *B.F.J.*, 1910, 112, 134, 149).

West London. Cocoa shell 18 %. The Public Analyst stated that he had found 7.05 % of fibre, 22.7 % of cocoa butter, and that the dry fat-free cocoa had 4.13 % of nitrogen. After much expert evidence the magistrate was not satisfied that 18 % of shell prevented it being cocoa of commerce, and dismissed the case (*Grocer*, 1910, Dec. 10).

Bury. Cocoa shell upwards of 25 %—probably 33 %. The Public Analyst gave evidence that cocoa beans naturally contained 8–13 % of shell which was ordinarily removed in the manufacture of cocoa. Fine £25 (*Grocer*, 1913, April 26 ; *B.F.J.*, 1913, 91).

Richmond. Arsenic $\frac{1}{10}$ grain per lb. The retailers had blended seven different cocoas, one of which contained $\frac{1}{10}$ grain arsenic per lb. The arsenic was traced to the carbonate of potash which had been used to make the cocoa soluble. The retailers were fined £2 and the manufacturers £20 and 50 guineas costs for aiding and abetting (*Grocer, P.J.*, 1922, Dec. 23 ; *B.F.J.*, 1922, 115 ; *Analyst*, 1923, 48, 67).

Reigate. Sugar 28 %, arrowroot 22 %. Fine 10s. (*Grocer*, 1925, Jan. 31).

PROSECUTION FOR COCOA AND MILK POWDER. *Brentford.* Cocoa 17 %, starch 17 %, sugar 66 %, and no milk. The Government analysts reported that milk solids, if present, did not amount to more than 1 %. Evidence was given that 1.1 % of condensed milk was added, equivalent to $3\frac{1}{2}$ % of milk powder. Costs 5 guineas (*Grocer*, 1905, Jan. 7, March 25 ; *B.F.J.*, 1905, 13).

CHOCOLATE

Chocolate consists of cocoa, sugar and flavouring materials, but there is a difference of opinion as to whether the use of starch and coconut stearine or other oils is legitimate ; on the question, see Booth (*Analyst*, 1909, 34, 401 ; *B.F.J.*, 1909, 142 ; *J.S.C.I.*, 1911, 235).

Samples of bar chocolate bought in Birmingham yielded 0.84–1.59 % of ash and 0.28–0.40 % of phosphate (P_2O_5). Chocolate creams had 0.35–0.58 % of ash and 0.11–0.16 % of P_2O_5 . The fat of twelve of the sixteen samples of both kinds examined had a B.-R. 40° figure of about 47, and a Valenta figure of about 113, showing the absence of foreign oils. There was also a badly rancid sample which contained coconut oil.

A particularly objectionable form of adulteration has been the coating of a sweetmeat containing glucose with paraffin wax, coloured with iron oxide, to prevent the absorption of moisture, and the sale of such an article as “chocolate chumps.” Paraffin wax is insoluble in the body, and in the Birmingham district a coroner’s jury returned a verdict on a child, as “Death from peritonitis, probably caused by paraffin in chocolate chumps.”

Booth, Cribb and Richards have given a paper on the composition and analysis of chocolate (*S.P.A.*, 1909, **34**, 134), and Wiseman has given a modification of the Gottlieb method for the determination of fat (*S.P.A.*, 1930, **55**, 684). Bywaters, Maggs and Pool have studied the determination of illipé butter (*S.P.A.*, 1927, **52**, 324); see also Knapp, Moss and Melley (*S.P.A.*, 1927, **52**, 452). For other methods of analysis see under cocoa (p. 333).

PROSECUTIONS. *Lambeth.* Coconut stearine 38.8 %. The magistrate, with some doubt and hesitation, dismissed the case. He said that there was no evidence of fraudulent addition or even inferior quality. Some vegetable oil in about the proportion present was necessary for its preparation as an article of commerce, and coconut stearine was not injurious to health (*F. & S.*, 1898, March 12).

Cardigan. White paste devoid of chocolate 55 %. It was stated that the article was labelled “chocolate cream” and that there was no standard. Dismissed (*B.F.J.*, 1903, 93).

Sunderland. Paraffin wax 4 %. Two vendors were each fined £5 (*P.J.*, 1903, Oct. 25).

London, Marlborough Street. Containing the acrid principles of cayenne pepper, and being injurious to health. They were sold for practical jokes. Fine £1 (*Grocer*, 1910, May 28; *B.F.J.*, 1910, 112).

PROSECUTION FOR PLAIN CHOCOLATE DROPS.

Westminster. Foreign starch 13.5 %, foreign fat 19.5 %. The fat-free cocoa matter contained not less than 27 % of cocoa shell or husk. Fine £1 (*B.F.J.*, 1909, 148).

PROSECUTION FOR CHOCOLATE MIDGETS. *St. Columb.*

Iron oxide 7 grains per lb. The Bench were of opinion that no definite standard had been placed before them and dismissed the case (*Grocer*, 1912, May 11; *B.F.J.*, 1912, 95).

PROSECUTION FOR CHOCOLATE ANIMALS. *Stanley.* Gelatin, maize starch, and ferric oxide present. Fine £2 (*Analyst*, 1924, **49**, 519; *B.F.J.*, 1924, 98).

ORDER FOR DESTRUCTION OF CHOCOLATE EASTER EGGS. *Liverpool.* They contained quartz, glass, lead, zinc, copper, and sawdust, and had been made from chocolate waste (*Analyst*, 1925, **50**, 238).

PROSECUTIONS FOR CHOCOLATE CHUMPS. *Birmingham.* Paraffin wax $2\frac{1}{2}$ %. Fine £5. Paraffin wax 3 %, $3\frac{1}{2}$ %, 5 %; each vendor was fined £2. The articles were certified to be injurious to health (1896 Report).

PROSECUTION FOR CHOCOLATE STICKS. *Durham.* Paraffin wax 6.23 %, and injurious to health. Medical evidence was given that the wax was an irritant, and might set up appendicitis. Each of the three partners was fined £5 (*P.J.*, 1902, Nov. 8).

PROSECUTION FOR CHOCOLATE CIGARS. *Glasgow.* Paraffin wax 4 %, to the prejudice of purchaser. Evidence was given that cocoa butter was used in the better qualities, but other fats for cheaper articles, and that both had been sold for years. It was held that evidence on injury to health was inadmissible under that section and case was dismissed (*British & Foreign Confectioner*, 1894, June 16).

PROSECUTION FOR "ALL FOOLS" CHOCOLATE. *East Ham.* Leather 46 %. The notice in the shop, "The greatest 'take in' ever invented into chocolate," was held to be insufficient. Fine £1 (*P.J.*, 1913, July 12; *B.F.J.*, 1913, 133).

PROSECUTIONS FOR CHOCOLATE LIQUEURS. *Manchester.* Selling spirits retail without a licence. The chocolates were in the form of bottles, and three samples contained liquids containing 8–12 % of proof spirit. Fine £3 (*P.J.*, 1911, April 22, 29).

St. Helens. A similar prosecution to the previous one. The liquid contained 2.45 % of proof spirit, or 1.03 % of the whole. As the proportion was under 2 %, the case was dismissed (*Analyst*, 1928, **53**, 280).

MILK CHOCOLATE

The introduction of milk into chocolate complicates the analysis by increasing the nitrogen, adding another sugar (lactose) and also butter-fat.

Five samples bought in Birmingham had 1.64–1.88 % of ash, 0.40–0.54 % of P_2O_5 , and 19.6–29.8 % of fat. The calculated milk solids-not-fat were 12.0–21.7 %, the milk fat 4.1–5.5 %, and the total milk solids 16.1–27.2 %.

Booth, Cribb and Richards have given analyses of twenty different makes of milk chocolate (*S.P.A.*, 1909, **34**, 146).

ANALYSIS. The milk solids-not-fat are obtained by multiplying the lactose by 24/13, and the milk fat by the following formula :—

$$\text{Milk fat, \%} = \frac{(\text{Reichert value} - 0.3) 100}{28 - 0.3}.$$

To obtain a solution suitable for the determination of the polarisation and reduction of the sugars present, add 90 ml. of water to 16 gm. dried fat-free chocolate, and leave over night. Then add 10 ml. of 2E.Pb \bar{A}_2 and filter. Mix 25 ml. of the filtrate with 25 ml. saturated solution of K₂SO₄; the filtrate will represent a 12 w/v solution.

PROSECUTIONS. *Marlborough Street.* Arrowroot starch 16 %, cocoa husk, coconut oil, and only a suspicion of cocoa. The maker gave evidence that the ingredients were: 2 cwt. of "mixture," 5 lb. nib cocoa, and some shell, and 5 lb. of dried separated milk. The magistrate said it was not chocolate, as there was not more than 2½ % of cocoa, and not milk chocolate, as the milk solids were less than 2½ %. Fine £5 and 10 guineas costs (*B.F.J.*, 1908, 141; *Grocer*, 1908, Aug. 8).

PROSECUTION FOR NUT MILK CHOCOLATE. *Birmingham.* Peanuts 12.9 %, sugar 78 %, and no chocolate. A card in the window described it as "Delicious imitation milk chocolate," but the word "imitation" was almost invisible, the other letters being fifty-six times larger. Fine £1 (*B.F.J.*, 1911, 10; *Grocer*, 1911, Jan. 14).

CHAPTER XXIII

AERATED DRINKS. DISTILLED WATER

Soda water. Potash water. Lithia water. Lemonade. Ginger beer. Ciderette. Distilled water.

THE term "aerated" is not strictly correct, as these drinks are charged with carbon dioxide, and not with air. One vendor indicated the freedom of his soda water from air by describing it as "apneumatic."

The chief questions that the analysis of such articles should answer are :—(1) Is the "soda" or other added constituents present in the proper proportion? (2) Is the article of potable quality, or are impurities present due to the use of an impure water, or defective cleanliness in the apparatus or bottles? (3) Is lead, copper, zinc or tin present, or phosphoric acid?

Three Birmingham samples from one company, sold as "Carbonised water only," contained 0.15–0.16 per 100,000 of copper, and 0.45–0.54 of lead. The alkalinity, due to the water used, was equivalent to 0.4 grain of sodium bicarbonate per pint.

According to the Government Reports, the percentages of adulteration of "aerated or mineral waters" in England and Wales were as follows :—1887–93, 28.2 %; 1902–13, 14.0 %; 1920–30, 5.4 %.

ANALYSIS. The determination of *alkalinity* and of *poisonous metals* has been given in previous chapters (pp. 67, 85, f). For the determination of *carbon dioxide*, see Stransky (*Analyst*, 1908, **33**, 103) and Cannizzaro (*Analyst*, 1923, **48**, 608).

Free and Albuminoid Ammonia. Elsdon and Evers have shown that carbon dioxide interferes with the colour of Nessler solution (*S.P.A.*, 1912, **37**, 173). Put 500 ml. of the well-shaken water into a 1,500 ml. distillation flask, and attach to a condenser which has been washed inside and out till the washings give no colour with Nessler solution. Ammonia on the outside of an unwashed condenser may seriously contaminate the distillate. Add sufficient $\text{N.H}_2\text{SO}_4$ to make acid, distil 50 ml. and reject the distillate. Add an equivalent quantity of N.NaOH , and about 2 ml. $\text{E.Na}_2\text{CO}_3$. Distil successive quantities of 50 ml. until the distillate gives no colour with 1 ml. Nessler solution. Add 50 ml. alkaline permanganate, and distil 50 ml. into a cylinder, or, with an impure water, into a graduated flask. Distil further quantities until the distillate is free, or practically free, from ammonia. At least two-thirds, and frequently all, the free ammonia comes over in the first cylinder.

The distillates, or fractions of distillate diluted to 50 ml., are compared with a series of standards made by adding standard AmCl solution (N/1703, 1 ml. = 0.00001 NH_3), to about 50 ml. water, adding 1 ml. Nessler solution, mixing, and allowing to stand a short time. It is not advisable to add the standard solution after the Nessler solution. All water used for dilution must be free from ammonia, and the cylinders must be colourless, and of similar diameters. If necessary, a correction for any ammonia present in the alkaline permanganate solution should be subtracted.

The alkaline permanganate is made by boiling 1 lb. of soda with 28 gm. of potassium permanganate and 4 litres of distilled water, and diluting when cold to $3\frac{1}{2}$ litres.

When water contains a volatile reducing substance like formic aldehyde, a yellow colour, due to the reduction of the Nessler solution, may be produced long after all the ammonia has been distilled over. When successive quantities of distillate give a similar colour with Nessler solution, alkaline permanganate solution should be added and the albuminoid ammonia determined. The *total* ammonia should be determined on another quantity, by adding alkaline permanganate solution before distillation. Free ammonia is obtained by subtracting the albuminoid ammonia from the total ammonia.

$$\text{NH}_3 \text{ per } 100,000 = \frac{\text{ml. AmCl solution} \times 100,000}{\text{ml. water taken.}}$$

Oxygen consumed. Into a 12-oz. stoppered flask put 10 ml. of about 9E. H_2SO_4 , which has been boiled with KMnO_4 , and 250 ml. of the water, put the flask into a water bath kept at 27°C ., preferably by means of a thermostat. After half an hour add 10 ml. N/80 KMnO_4 solution, and heat at 27°C . for three hours. Then add 0.5 ml. E.KI solution cool and titrate with 0.25 w/v solution of $\text{Na}_2\text{S}_2\text{O}_3$, using starch solution towards the end of the titration. Number of ml. used = B.

To 250 ml. of distilled water, add 10 ml. each of the H_2SO_4 and KMnO_4 solutions, and 0.5 ml. of E.KI solution at once. Titrate with the $\text{Na}_2\text{S}_2\text{O}_3$, ml. used = A.

If on heating the purple colour fades rapidly, more KMnO_4 solution should be added, as the reaction is not complete unless there is an excess of about 2 ml. An alternative is to use less water and make the volume up to 250 ml. with distilled water free from organic matter. If "C" ml. of KMnO_4 are used in the determination :—

$$\begin{aligned} \text{Oxygen consumed per } 100,000 &= \frac{(\text{CA} - 10 \text{ B}) 0.0001 \times 100,000}{\text{A} \times \text{ml. water taken}} \\ \text{or } &\frac{(0.1 \text{ A} - \text{B}) 100}{\text{A} \times \text{ml. water}} \end{aligned}$$

Experiments have proved that with most waters there is little difference between four hours' heating and the more convenient time of three hours given above. As the KMnO_4 solution is used to standardise the more unstable $\text{Na}_2\text{S}_2\text{O}_3$ solution, it is not advisable to heat the distilled water and the KMnO_4 before titrating, as is recommended in some books. Also, that method will give the *difference* between the amount of organic matter in the distilled water and the water examined, instead of the *total* amount.

Nitrites. Add 2 ml. Greiss-Ilosvay reagent to 50 ml. water in Nessler cylinder, and prepare standards from standard NaNO_2 solution, containing 1 of nitrous nitrogen per million (conveniently made by diluting 1 ml. of 0.5 w/v solution of NaNO_2 to 1,000 ml. with nitrite-free water), and allow to stand an hour or more.

Nitrous nitrogen per 100,000 = ml. of standard to match 50 ml. water $\times 0.002$.

Nitrates by Crum method. Treat the total solids of the water with a few ml. $3\text{E.H}_2\text{SO}_4$; after standing a few minutes transfer to a nitrometer; if necessary use a *few* ml. water to wash out the dish. Pour into the cup of the nitrometer a volume of H_2SO_4 (free from nitrate) about one and a half times the volume of the acid liquid previously added. Run the acid through the tap of the nitrometer, taking care not to introduce air, then shake the acid and the mercury together till there is no further increase in the volume of the gas. When cold adjust the level, and measure the volume of the gas.

Nitrogen as nitrates and nitrites per 100,000 = $1.1 \times$ ml. NO from 50 ml. water.

With high chlorides HCl may be given off; if this be suspected, carefully introduce a drop of water and note any reduction of volume. The determination is conveniently made with a nitrometer which is open at the bottom, contained in a special mahogany trough.

SODA WATER

Until 1898 this article was in the B.P., and contained 30 grains of sodium bicarbonate per pint. "Soda water" is now included in the B.P. Codex, which orders 3 grains of sodium bicarbonate, $1\frac{1}{2}$ grains of sodium chloride, and $\frac{1}{2}$ grain of sodium sulphate in a pint of distilled water.

Owing to the great alkalinity of the B.P. article making it unsuitable for use as a beverage, a practice arose of selling carbonated water with little or no added sodium bicarbonate as "soda water"; prosecutions were instituted, and the B.P. standard for the *drug* was incorrectly applied to the article sold by publicans as a *beverage*.

ALKALINITY OF SODA WATER, 1911-5 (Seventy-five samples)

Total alkalinity, expressed
as grains of sodium

bicarbonate per pint	. 0.4-	3.0-	6.0-	9.0-	12.0-	14.4	15.1-	33.5	Total.
Percentage of samples	. 44	15	18	7	7		9		100

It will be seen that the total alkalinity, expressed as sodium bicarbonate, of nearly half of them was under 3 grains per pint, and that in only 9 % did it exceed 15 grains ; only three samples approached the B.P. standard. One sample was labelled " Superior Carbonated Water, commonly called Soda Water." One firm sold two different articles, one labelled 5 grains, and the other 30 grains, per pint.

If soda water were always prepared with distilled water, the above figures would represent the amounts of *sodium* bicarbonate present, but in nearly every case, the water used contains more or less *calcium* bicarbonate. The figures, therefore, are the alkalinities due to both sodium and calcium bicarbonate, expressed as sodium bicarbonate.

In some samples no sodium bicarbonate was present, and in others its presence was problematic. The following table illustrates this :—

ANALYSES OF WATER AND SODA WATER

GRAINS PER PINT.	PUBLIC WATER SUPPLY.			SODA WATER.				
	A	B	C	D	E	F	G	H
Salt (calculated from chlorine)	0.1	0.4	0.5	0.2	0.4	3.2	7.2	0.1
Calcium carbonate (calculated from total alkalinity)	. 0.2	1.3	1.4	0.3	1.2	1.2	8.3	19.7
Other solids (by difference)	. 0.2	1.3	2.5	0.1	2.0	13.1	5.3	2.5
Total solids	. 0.5	3.0	4.4	0.6	3.6	17.5	20.8	22.3
Sodium bicarbonate (calculated from total alkalinity)	. 0.3	2.1	2.4	0.5	2.0	1.9	13.8	33.1

The nearness of the figures for water A and soda water D, and of water B and soda water E, suggests that, in each case, *all* the alkalinity was due to the original water.

Sample F claimed to be made from artesian well water, and the 13.1 grains of " other solids " (which might be either calcium or magnesium, sulphate or nitrate), support that statement, the alkalinity being due to calcium bicarbonate.

Salt had probably been added to G, and the 5.3 grains of " other solids " suggests that a fair proportion of calcium bicarbonate was present.

Sample H alone was approximately of B.P. strength, and *sodium* carbonate (which is formed from the bicarbonate on drying), and not calcium carbonate, would be weighed in the total solids. Correcting for this the " other solids " would be 1.3, and not 2.5.

In the next table analyses are given to illustrate the potability of the waters :—

PARTS PER 100,000.	J	K	L	M	N	O	P
<i>Chemical Results.</i>							
Ammonia, free	0.003	0.003	0.002	0.006	0.002	0.004	0.009
„ albuminoid	0.004	0.003	0.013	0.022	0.006	0.008	0.015
Nitrogen in nitrates . . .	2.9	0.4	0.3	0	0	0	3.5
Oxygen consumed (three hours)	—	—	—	0.51	—	0.68	—
Chlorine, combined	6.5	2.6	22.7	0.8	1.2	39.8	54.8
Solids, other than carbonate or chloride	35	23	20	10	5	38	69
Loss of solids on ignition .	—	—	—	12	—	42	—
<i>Bacteriological Results.</i>							
Glucose taurocholate, 50 ml.	0	0	+	+	0	+	+
„ „ 10 ml.	0	0	+	+	0	+	+
„ „ 1 ml.	0	0	0	0	0	+	0
Rebipolagar, two days, 1 ml.	—	—	—	1,000	—	34	—
Jelly, three days, 1 ml.	4	3	540	35,000	70	350	1,000
Container	Sy.	Sy.	Ser.	Ser.	Cd.	Cd.	Ck.

The first two samples were taken from syphons, and the ammonias and bacteriological results were satisfactory. The high nitrates and chlorine of J, however, arouse suspicion, unless the water used was from a deep well. L was taken from a screw-stoppered bottle, from the same maker as K, but the results were much less satisfactory ; they also indicate added salt. M, from another screw-stoppered bottle, was of very unsatisfactory quality, probably from the use of a dirty bottle. Of the two bottles closed by a glass marble (Codd), N gave low figures ; the jelly count (70) appears reasonable for water that is not from a syphon. The other one gave bad results, due either to the use of an impure water or lack of cleanliness.

The last sample, from a corked bottle, in addition to being high in ammonias, nitrates and micro-organisms, had enough “ other solids ” to condemn it as too hard for drinking purposes.

The work of Elsdon and Evers (*S.P.A.*, 1912, **37**, 395) shows that on keeping the number of micro-organisms, particularly those growing on jelly, diminishes. A low bacterial count, therefore, does not necessarily mean that the original water was pure.

It will be seen that caution is necessary in judging of the potability of a soda water. High solids and alkalinity ought to be due to added sodium carbonate, but may not be. High chlorine may be due to added salt. Impurity of the sodium bicarbonate may cause an increase in the free ammonia, and bacterial counts depend on the age of the water. The most useful tests for impurity are probably those for albuminoid ammonia, oxygen consumed, and the glucose taurocholate tests.

The contact of soda water with lead pipes is a potential source of lead poisoning. In 1907 the Manchester Public Analyst found 3 grains of lead per gallon in soda water that had been in a machine

all night, and in the same year an inquest followed a death due to soda water containing 7 grains per gallon. At least half of the samples of fountain soda water examined by the Queensland Government Analyst contained over $\frac{1}{20}$ grain per gallon, which is considered a dangerous amount (*Analyst*, 1928, 53, 39).

Of the fifty-three samples of Birmingham soda water examined 1911-2, forty-three contained less than 1 part of lead per million, five contained 1-3 parts, and five 4-5.4 parts. Most of the higher figures were due to water from syphons. High lead figures occurred both in waters high in alkalinity and in those which were low.

The head of one of the syphons was soaked for a week in soda water having an alkalinity equal to 2.6 grains of sodium bicarbonate per pint. The lead increased from 0.4 to 1.0 part per million, but 0.3 part of copper disappeared.

Copper was determined in thirty-three samples; twenty-eight of them had less than 1.0 part per million, the other five had 1.0-1.5 parts. In two cases tin was detected, 0.4 and 0.5 per million being present. Again, the higher figures were in water from syphons. Only one sample contained zinc, 500 parts per million being found.

No prosecutions for soda water have been instituted in Birmingham, but in 1911 representatives of the mineral water trade were called to a meeting and cautioned by the Chairman of the Public Health Committee. The position taken was that soda water should be made from clean water, be put in clean bottles, be free from lead, and contain an appreciable amount of sodium bicarbonate. Of the fifty-seven samples examined 1911-5, no less than forty-three were condemned for one or more defects.

PROSECUTIONS. *Londonderry.* Lead $\frac{1}{2}$ grain per gallon. The Government analysts reported that it contained $\frac{1}{100}$ grain of tin and lead in 10 oz., the latter being present to the extent of only a trace. Fine £1 (*P.J.*, 1893, June 17).

Dumfries. No bicarbonate of soda. For the chemist who sold it, the plea was made that the B.P. article contained so much alkali, that it was not only disagreeable to the taste, but also injurious, when used as a beverage. Fine £2 (*F. & S.*, 1894, Nov. 10).

Peterhead. Brewer prosecuted for a sample entirely devoid of carbonate of soda, and containing traces of copper. Fine £3 (*B.F.J.*, 1902, 202).

Rochdale. Sodium bicarbonate not more than 1 grain per pint, instead of 8 to 10. The article was bought at an hotel. A mineral water manufacturer gave evidence that reputable firms put in 10 grains per pint on an average, and that bought samples contained 3 grains or more. The makers of the article explained that the deficiency was due to bad mixing, as 10 grains per pint should have been there. Fine 10s. 6d. (*B.F.J.*, 1909, 194).

POTASH WATER

Until 1898 this article was in the B.P., and contained 30 grains of potassium bicarbonate per pint. It is in the B.P. Codex, which requires 15 grains per pint.

PROSECUTIONS. *Swindon.* Carbonated water, destitute of potash. The syphon from which the sample was taken was labelled "Potash water." The vendor was fined 1s. and costs. The manufacturer of the water was prosecuted under the Merchandise Marks Act for applying a false trade description to the above water. The defendant suggested that a potash water label had been put on a syphon of carbonated water, as his practice was to put 8 grains of potash per pint. Fined 10s. and costs (*C. & D.*, 1899, Dec. 2 ; *F. & S.*, 1899, Dec. 9).

Swindon. Carbonated water, destitute of bicarbonate of potash. The Public Analyst admitted he had only looked for bicarbonate of potash. An analysis made for the vendor by another public analyst indicated 6 grains of bicarbonate of potash per pint, and 25 grains of other solids. Later, after a joint examination, the two analysts agreed that the sample was practically destitute of bicarbonate of potash, but contained 5 grains of bicarbonate of soda per pint. Fine 1s. (*B.F.J.*, 1899, 379 ; 1900, 26).

Margate. Aerated water containing no potash. Fine £1 and costs. A mineral water manufacturer was fined £10 and costs for a similar offence (*B.F.J.*, 1902, 229).

Bournemouth. Bicarbonate of potash 3·7 grains per pint, instead of at least 10 grains. The magistrates decided there was no standard and dismissed the case (*B.F.J.*, 1904, 238).

LITHIA WATER

Until 1898 this article was in the B.P., and contained 10 grains of lithium carbonate per pint. It is in the B.P. Codex, unaltered in strength.

PROSECUTIONS. *Margate.* Aerated water containing no lithia. Fine £1. A mineral water manufacturer was fined £10 for a similar offence (*B.F.J.*, 1902, 229).

Bournemouth. Total solids 6·4 grains per pint, of which only 3·5 grains was carbonate of lithia. Medical evidence indicated that lithia water containing less than 5 grains per pint was rarely prescribed, and that the 10 grains standard was satisfactory. The defence was that lithia water was often taken as a beverage, that 3·5 grains per pint had medicinal value, and that natural lithia waters did not contain more than 2 grains. Owing to the absence of a standard the magistrates dismissed this case, but inflicted a fine of 10s. for a sample which contained only $\frac{1}{2}$ grain per pint.

In other cases when lithia was absent the fine was £1. The defendants included a grocer and an hotel keeper (*B.F.J.*, 1904, 236).

Bournemouth. Lithium carbonate 1·2 grains per pint, instead of 10 grains. The vendor was summoned for selling a “food,” but the Bench amended it to “drug.” After conflicting medical evidence, the magistrates were evenly divided in opinion, and when the case was reheard, a majority of the magistrates dismissed it (*P.J.*, 1908, Oct. 17, 31; *B.F.J.*, 1908, 174, 193).

LEMONADE

The amount of solid matter in carbonated lemonade examined in Birmingham, mostly in 1915, was as follows :—

SOLID MATTER IN LEMONADE

Solids w/v	1·5–	3·0–	5·0–	7·0–8·6	Total.
No. samples	6	7	3	3	19

The acidity, determined by phenol phthalein after boiling, gave the following range :—

ACIDITY OF LEMONADE

Acidity as citric acid w/v.	0·06–	0·10–0·17	0·41	Total.
No. samples	11	6	1	18

Of the sixteen samples tested for salicylic acid, only two contained it, the amount present being 40 and 10 parts per million. Fifteen samples were tested for saccharin, and it was detected in eleven of them. Five determinations indicated 11 to 70 parts per million. The presence of saccharin in lemonade raised a difficult question. While there is no doubt its presence was a recent addition, and therefore unjustified without declaration, the large proportion of samples containing it indicated that prosecution, particularly in war-time when sugar was scarce, was inadvisable. The question was more a national one than a municipal one, and the attention of the Ministry of Health was called to the subject.

The use of phosphoric acid for making lemonade is unauthorised and undesirable.

PROSECUTIONS. *Otley.* Lead $1\frac{1}{4}$ grains per gallon. The defendant’s analyst found 0·05 grain, and the Government analysts 0·30 grain, per gallon. The difference in the analyses was probably due to one of the three bottles bought being sent to each analyst without any mixing before division. The case was dismissed (*Analyst*, 1884, 9, 194).

Aberdeen. Lead 0·2 grain per gallon. The Government analysts found no lead, but “a minute trace of copper.” Correspondence

between the analysts indicated agreement that about 0.2 grain of metal was present, and that the point at issue was the nature of the metal. The case was dismissed (*B.F.J.*, 1901, 31, 69, 290).

The defendant afterwards sued the Public Analyst for £1,000 damages for injury due to the Analyst's alleged carelessness and lack of skill, but the action was dismissed (*B.F.J.*, 1901, 175).

Hayle. Copper 4 grains per gallon. Fine 2s. 6d. (*P.J.*, 1906, March 31).

Kingston. No lemon juice, but dilute solutions of sugar, flavoured (one artificially coloured), and acidified with phosphoric acid. One was labelled "Sparkling lemonade," the other "Still lemonade." In one case there was also a notice displayed "Made from Messina lemons." For the defence it was stated that phosphoric acid was preferred to citric and tartaric acids, as the latter might contain lead, and that it was flavoured with an extract made from the peel of Messina lemons. The retailers were fined 10s. each, and the manufacturer paid £41 in fines and costs for offences under the Food and Drugs Act, the Merchandise Marks Act, and for aiding and abetting (*Grocer*, 1921, July 2; *B.F.J.*, 1921, 74).

Blackpool. Entirely devoid of lemon, being composed of 97 % of water, 2.4 % of sugar and 0.6 % of tartaric acid. Fine 10s. (*B.F.J.*, 1926, 75).

London, South-Western. Soluble lead salt equivalent to 26 parts of metallic lead per million. The lemonade was being sold from a glass vessel, through a lead pipe syphon. Vendor was ordered to pay 10s. costs. Another vendor was fined 10s. for 8 parts per million (*B.F.J.*, 1927, 86).

GINGER BEER

PROSECUTIONS. *Camelford.* Copper 0.0014 grain per gallon. The defendant admitted the offence, and suggested that copper filings had been left in the cauldron, when recently repaired. Fine 10s. and costs (*B.F.J.*, 1904, 261).

Tooting. Metallic lead 1.54 grains per gallon, and injurious to health. The ginger beer had been drawn from the cellar through a lead pipe. The Medical Officer of Health said that a death from lead poisoning, attributed to ginger beer, had occurred, and that notices had been sent to vendors in the Borough, warning them against the use of lead pipes. Vendor paid 12s. 6d. costs (*P.J.*, 1909, Nov. 20; *B.F.J.*, 1909, 206).

London, South-Western. Soluble lead salts, due to lead pipes being used to draw the ginger beer from the cellar, instead of having the cask on the counter. It was stated that out of ninety-seven public-houses visited by the Council's inspectors, ginger beer was taken from the barrel in seventy-nine cases. The magistrate, from

his reading of the section, assumed that deliberate admixture must be proved. He dismissed the summons on the ground the vendor did not knowingly mix anything with the ginger beer, and that therefore he had no guilty knowledge (*B.F.J.*, 1912, 158).

CIDERETTE

Louth. Salicylic acid 3·5 grains per pint. Fine 5s. (*B.F.J.*, 1914, 157).

DISTILLED WATER

In spite of objections, it has been ruled in prosecutions that distilled water is a "drug." In 1910, Cowrie reported on seven samples of commercial distilled water (*P.J.*, Jan. 8). There was a slight opalescence and fluff in five of them, which was enough to condemn them. The amount of solid residue from 100 ml. was 0·001–0·0046, and was below the B.P. limit of 0·005, a limit, however, which would pass much Birmingham undistilled Welsh tap water. Only one sample had too much ammonia, eight times the limit being present. It had been made by continuous distillation, and hypodermic solutions prepared with it were inferior. Three samples contained lead, 0·5–1·0 per 100,000, attributed to the container. Storage in dark glass bottles, which are generally free from lead, is recommended, and it is remarked that ammonia promotes the solution of lead. When bought for analysis glass-stoppered bottles should be used.

PROSECUTIONS. *Marylebone.* Excess of residue and organic matter, and 3·5 parts of ammonia per million. A very dirty sample, full of mouldy growths. The defendant explained that he thought the water was required for photographic purposes. The objection that distilled water was neither a food nor a drug was overruled by the magistrate, as it was largely used in making up medicines. Fine 10s. (*P.J.*, 1906, June 20 ; *B.F.J.*, 1906, 50).

Hampstead. Dirt or extraneous matter, consisting of vegetable hairs and carbonate of lime, $5\frac{1}{2}$ grains per gallon. The Government analysts found 4·67 grain per gallon of mineral matter, chiefly calcium carbonate, and 0·37 grain per gallon of organic matter, including fragments of cork and decayed cork. It could not properly be described as distilled water. For the defence it was suggested that the impurity was due to the use of old and defective corks for the sample bottles and that the absence of chlorides showed it was not tap water. The magistrates decided that the article was a drug, but considered the matter was too doubtful for a conviction and dismissed the case (*P.J.*, 1909, March 6).

CHAPTER XXIV

ALCOHOL

Expression, and calculation, in various terms. Determination, spirits, beer, tinctures. Methyiated spirit. Iso-propyl alcohol.

THE legal definitions of the strength of alcoholic drinks are expressed in terms of "proof spirit," which itself has been defined as "that which at the temperature of 51° F. weighs exactly $\frac{1}{3}$ ths of an equal measure of distilled water" (56 George III., c. 140).

The term "proof" is derived from the old smuggling days. After a cask of spirit had been seized by the coastguard, the strength of the spirit was roughly tested by pouring a little on gunpowder and lighting the spirit. If, when the spirit had burned off, the gunpowder fired, the sample was "over proof." If, however, the amount of water present in the spirit was sufficient to make the gunpowder too damp to fire, the sample was "under proof."

The strength of mixtures of alcohol and water may also be expressed as percentage by weight (w/w), by volume (v/v), or as gms. per 100 ml. (w/v). The table below gives particulars of typical spirits, taken or calculated from Thorpe's Tables ("Alcoholometric Tables," by Sir E. Thorpe):—

STRENGTH OF SPIRITS

Spirit.	Absolute alcohol.	Alcohol 1932 B.P.	Proof spirit.	25° Under proof.	35° Under proof.
Specific gravity, 60°/60° F.	0.79359	0.8159	0.91976	0.94716	0.95608
Proof spirit, v/v . . .	175.35	166.6	100.00	75.00	65.00
Absolute alcohol, w/w . . .	100.00	92.41	49.28	35.92	30.85
Water, w/w	0	7.59	50.72	64.08	69.15
Absolute alcohol, v/v . . .	100.00	95.00	57.10	42.85	37.15
Water, v/v or w/v	0	6.19	46.66	60.70	66.12
Absolute alcohol, w/v . . .	79.359	75.40	45.32	34.02	29.50

The above table illustrates the use of the term "proof." Spirits containing 75 v/v and 65 v/v of proof spirit are described as "25° under proof," and "35° under proof," respectively. The abnormality of the strength of alcohol being 166.6 v/v proof spirit, is avoided in practice by such spirit being described as "66.6°" over proof." If 100 ml. of it be diluted to 166.6 ml., at the same temperature, proof spirit will result. It should be noticed that while 100 ml. of proof spirit contains 57.1 ml. of absolute alcohol, it also contains 46.66 ml. of water, and not 42.9 ml., as would be expected. This is due to the mutual penetration of the alcohol and water producing a contraction of 3.76 ml.

Proof spirit is always expressed as percentages by volume, but unfortunately some writers use the term "percentage of alcohol" without any indication to show if percentage is by weight or volume: the above table shows that the differences between the two statements may be considerable.

In the calculation of the percentage volume of excess water present in an adulterated spirit, any of the volume standards may be used, but not the percentage by weight. This may be illustrated by a Scotch whisky which was 56.77° U.P., instead of 35° U.P.

CALCULATION OF ADULTERATION OF WHISKY

Alcohol expressed as :	Absolute alcohol. v/v.	Absolute alcohol. w/v.	Proof spirit. v/v.
Amount present .	32.47	25.78	56.77
Excess water, v/v. .	$\frac{(37.15-32.47)100}{37.15}$	$\frac{(29.50-25.78)100}{29.50}$	$\frac{(65.0-56.77)100}{65}$

Each of the calculations indicates that the sample of Scotch whisky contained 12.6 v/v of excess water as compared with the legal limit of 35° U.P.

The determination of the sp. gr. at 60°/60° F. of liquids which consist of alcohol and water *only*, and reference to an alcohol table, will give the strength.

The strength of alcohol expressed in one form can be converted into another by the use of the following equations, which are based on Thorpe's tables. As the co-efficients of expansion of alcohol and water are different, and as the strengths of proof spirit in these tables were obtained by multiplying alcohol, v/v, at 50° F. by 1.7535, the factors containing these forms are only exact at *one* strength. Proof spirit has been taken as a mean strength, and the error with other strengths is small. The factor in the table for proof strength is 1.751; for absolute alcohol, it should be 1.7535, and for 25 w/v, it should be 1.7482 (see Liversseege, *S.P.A.*, 1931, 56, 529).

CONVERSION OF ALCOHOLIC STRENGTHS

Absolute alcohol, w/w	= $\frac{0.7940 \text{ v/v}}{\text{sp. gr.}}$	= $\frac{\text{w/v}}{\text{sp. gr.}}$	= $\frac{0.4533\text{P}}{\text{sp. gr.}}$
Absolute alcohol, v/v	= $1.259 \text{ w/w} \times \text{sp. gr.}$	= 1.259 w/v	= 0.5711P
Absolute alcohol, w/v	= $\text{sp. gr.} \times \text{w/w}$	= 0.7940 v/v	= 0.4533P
Proof spirit, v/v	= $2.206 \text{ w/w} \times \text{sp. gr.}$	= 1.751 v/v	= 2.206 w/v.

Absolute alcohol, w/v, indicates the number of gms. in a volume equal to that occupied by 100 gm. of water at 60° F.

If the sp. gr. of a spirit solution is taken at a temperature other than 60° F. it is necessary to correct the result to that temperature, and the correction will depend on the strength (or sp. gr.) of the spirit. The classical tables of Gilpin (*Trans. Royal Soc.*, 1792, 425) give the sp. gr. of forty strengths of alcohol at various temperatures.

Squibb (*Ephemeris*, 1884, May) has also published tables. Calculations from these results were plotted by the writer, and a table of corrections prepared (*S.P.A.*, 1897, **22**, 154). This table, which is given in the Appendix (p. 362), has been annexed by several writers without any acknowledgment. Richmond has compiled a table for the same purpose to be used between 10° and 25° C. (*S.P.A.*, 1920, **45**, 222).

Owing to the high price of spirits, the samples submitted to the Public Analyst are often small, and as the determination of sp. gr. may have to be made on 25 ml., great care is necessary for adulterated samples. The writer prefers to use a 25 ml. pycnometer, with a mark on the neck, into which a delicate Fahrenheit thermometer 5 mm. in diameter can just be inserted. A range of 55° to 65° F. is sufficient. The scale is graduated in fifths of a degree, and its length is about 66 mm. The scale should, if necessary, be corrected for any rise of zero in the thermometer, and correction must be made for any error in the pycnometer, reference to which has been made previously (p. 105).

Determination of Alcohol in Spirits. Fill a 25-ml. pycnometer just above the mark with the spirit at about 60° F., stir with the thermometer, and read when the temperature is constant. Adjust the volume, using the thermometer as a rod, without handling the pycnometer. Weigh, add more spirit and repeat the operation. If the two corrected sp. gr. results are discordant, repeat again.

Empty the pycnometer into an 80 ml. Wurtz flask, wash out with 10 ml. of water, attach to vertical spiral condenser, having an adaptor passing into the neck of the pycnometer. Distill a little more than 25 ml., shake and weigh (A). Take the temperature, adjust the volume and weigh (B). Add a drop or two of water, shake, weigh (C). Take temperature, adjust volume and weigh (D). Calculate sp. gr. from A and B, and obtain the respective strengths (v/v) of proof spirit.

Calculation :—

(1) $25 : A - B :: v/v \text{ indicated by B} : (\text{correction to be added to } v/v \text{ indicated by B}).$

(2) $25 : (A - B) + (C - D) :: v/v \text{ indicated by D} : (\text{correction to be added to } v/v \text{ indicated by D}).$

Taking 25, instead of actual weight, introduces an error of 0.01 v/v. A similar error is introduced by using the sum of the two corrections in (2) instead of each separately.

The above procedure is somewhat complicated, but it gives confirmatory figures when the quantity of the sample does not permit of a duplicate analysis. The efficiency of the condenser, etc., should be tested by taking the sp. gr. of a spirit before and after distillation. Experiments have shown that all the alcohol present in proof spirit distills over in the first half of the distillate.

Obscuration. Alcoholic drinks contain more or less soluble matter in addition to alcohol and water. Solid soluble matter, or carbon dioxide present in quantity, will increase the sp. gr., and therefore the alcoholic content indicated by the sp. gr. of the drink itself will be too low. The difference between the apparent and real strengths is called "obscuration." On the other hand, if ether or similar light liquid be present, the alcoholic indication will be too high.

If the quantitative effect of the solid matter on the gravity is known, the sp. gr. can be corrected by subtracting the product of the factor by the amount of solid matter. This factor has been determined by the writer by evaporating 200 ml. or more of a spirit to about 20 ml., filtering out any particles of solid, putting the filtrate in a 25-ml. pycnometer, diluting to the mark at about 60° F. and taking the sp. gr. The liquid was then evaporated and the solid matter dried and weighed. Irish whisky, Scotch whisky, gin, rum, and brandy, all gave similar results, *viz.*, that 1 w/v of solid extract increased the sp. gr. by 0.004.

The routine determination of the alcoholic strength of spirits may be much facilitated by taking the sp. gr. with a special hydrometer, determining the solid extract, and using the above correction for obscuration. The special hydrometer was made for me by Bailey, of Bennett's Hill, Birmingham (who also made the above-mentioned thermometer). It had a range from 0.930–0.960, the length of the scale being about 74 mm.; the diameter of the cylindrical bulb was about 22 mm., and the total volume about 21 ml. Using a 50-ml. Nessler cylinder, having an internal diameter about 27 mm., the sp. gr. of small quantities of spirits could be taken with reasonable accuracy for genuine samples. If adulterated, the indication was confirmed by the pycnometer.

A whisky, which contained 0.26 w/v of solid extract, may be taken as an example of the corrections for temperature and obscuration :—

Sp. gr. at 57° F.	0.9551	= 66.2 v/v proof spirit.
Temperature correction = 0.00035		
(60–57)	0.0010	
	<hr style="width: 50%; margin: 0 auto;"/>	

Sp. gr. at 60° F.	0.9541	= 67.4 v/v proof spirit.
Correction for extract = 0.26 ×		
0.0004	0.0010	
	<hr style="width: 50%; margin: 0 auto;"/>	

Sp. gr. of alcohol in whisky	0.9531	= 68.6 v/v proof spirit.
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Determination of Alcohol in Beer, etc. Most of the carbon dioxide, with which beer is usually saturated, should be removed by tossing between two beakers, to avoid its interference with the sp. gr. The acidity of a sample of beer determined by NaOH using phenol

phthalein as an indicator was 4.2 N.v/v; after tossing ten times the acidity was 2.7 N.v/v, after twenty times 2.4, and after thirty and fifty times it was 2.2. Tossing thirty times is apparently sufficient.

Distillation Method. Determine sp. gr. in 100 ml. pyknometer as near 60° F. as convenient (1° F. = 0.00013 sp. gr.). Empty into 250-ml. Wurtz flask and wash out with 10 ml. water. Add a little broken pot and distill about 70 ml. into the pyknometer, an adaptor being used. Dilute distillate to nearly 100 ml., adjust temperature to about 60° F., add water to the mark and weigh. Calculate sp. gr. and refer to alcohol table. The distillate may be titrated with N/10 NaOH and phenol phthalein. The distillate from a sour beer may contain acetic acid, which would increase the sp. gr.

If the original gravity is also required, the residue in the flask should also be diluted to 100 ml. at 60° and weighed.

Evaporation Method. Determine sp. gr. in 50 ml. pyknometer as near 60° F. as convenient. Empty the pyknometer into a porcelain dish and wash out with 10 ml. water. Evaporate to about half on water bath, return to pyknometer and wash out dish with water to make nearly 50 ml. Adjust temperature to about 60° F., add water to mark, and weigh.

Sp. gr. of alcohol = sp. gr. of beer — sp. gr. of extract — 0.00016.

The calculation for a sample of ale was :—

Sp. gr. of alcohol = 1.0093 — 1.0186 — 0.00016 = 0.99054. The result obtained by distillation was 0.99060.

The correction of 0.00016 is given in an elaborate report by H. T. Brown (*J. Inst. Brew.*, 1914, 662; *Analyst*, 1915, **40**, 124); he attributes it to the greater contraction that occurs when alcohol is mixed with sugar solutions than when it is mixed with water.

Determination of Alcohol in Tinctures. A. H. Allen (*S.P.A.*, 1879, **4**, 102) divided the tinctures into three classes :—(1) Those in which the alcohol could be satisfactorily separated by simple distillation, such as opium and nux vomica. (2) Those which required making alkaline with NaOH before distillation, such as ferric acetate. (3) Those which yield essential oils as well as alcohol on distillation. For the latter he proposed the following method. Dilute 50 ml. to about 350 ml., add a few drops of a strong solution of calcium chloride, then solution of sodium phosphate; shake and dilute to 400 ml., or 500 ml. if the tincture had been prepared with rectified spirit. The precipitated calcium phosphate, with the entangled essential oil, is then filtered out, and 250 ml. of the filtrate is distilled.

In some cases, such as paregoric, the addition of magnesium carbonate to the diluted tincture may be advisable, followed by filtration and distillation. To tincture of iodine, sodium thiosulphate and NaOH should be added before distillation. Sal volatile should be

diluted, treated with strong alum solution, acidified with H_2SO_4 , and tossed to remove carbon dioxide before distillation.

Thorpe and Holmes (*Proc. Chem. Soc.*, 1903, 13; *Analyst*, 1903, **28**, 110) have given a method for determining alcohol in the presence of ether, essential oils, etc. The tincture is shaken with salt solution and petroleum ether, and after separation of the petroleum ether solution containing the essential oil, etc., the salt solution is distilled. E. A. Mann (*J.S.C.I.*, 1925, **24**, 1284) has published comparative experiments on the methods of Allen, Thorpe and Holmes, and an American one. Richmond (*S.P.A.*, 1899, **24**, 201) advises shaking with ether-saturated water to determine alcohol in the presence of ether and petroleum ether.

Carl Jungk Method. This method has been modified by the writer (*C. & D.*, 1891, March 14) as follows:—Put 50 grain-measures of the tincture into a stoppered cylinder and add 200 grain-measures of methylated ether (sp. gr. 0.717), shake, allow to settle for five minutes and measure the lower layer.

TABLE FOR CARL JUNGK METHOD

Lower layer, grain-measures	. 2.5	5.0	7.5	10.0	12.5	15.0	17.5	20.0	22.5
Proof spirit, v/v	. 122	119	116	113	110	107	103	100	96
Lower layer, grain-measures	. 25.0	27.5	30.0	32.5	35.0	37.5	40.0	42.5	45.0
Proof spirit, v/v	. 92	88	84	79	74	68	62	56	50

If care is taken to use dry cylinders, to give time for the tincture to run down the sides of the cylinder and obtain the correct volume before adding the ether, the method will give useful comparative results, while using little of the tincture. A 25-ml. cylinder may be used with proportionate quantities, but usually the graduations of grain-measures are closer than those of ml. Glycerin must not exceed a small proportion.

Calculation from Solids. If, in the preparation of a particular tincture, the spirit used has sp. gr. " S_1 " (equal to " A_1 " w/v alcohol), and the tincture has sp. gr. " S_2 ," and contains " Ew " w/v of solid matter, and $\frac{S_2 - S_1}{Ew} = K$, then $S_1 = S_2 - K Ew$.

If, as in whisky, the volume occupied by Ew is very small, S_1 , so calculated, indicates the alcoholic strength " A_2 " w/v of the tincture. Camphor, on the other hand, adds its volume without contraction, when dissolved in spirit, and in this case A_1 requires reduction according to the volume of camphor present, then

$$A_2 = \frac{A_1 (100 - Ew)}{100}.$$

These are extreme cases, and usually a gm. of solids will not have the volume of a ml. in solution, and Ew in the last equation will require multiplication by a factor depending on the drug used.

Concrete examples of the method are given later under tincture of iodine, tincture of myrrh, and compound tincture of benzoin.

A table giving the w/v of alcohol in relation to sp. gr. is given in the Appendix (p. 362).

Methylated Spirit. Spirits have sometimes to be tested for the presence of methylated spirit, and the following tests have been tried by the writer :—(1) Potash test. Shake 1 ml. of 10E.KOH with 10 ml. of the spirit in a small stoppered bottle. A yellow colour begins to appear at once, but is better observed after standing overnight. (2) Ashby's test (*S.P.A.*, 1894, **19**, 268). To 5 ml. of spirit add an equal volume of a freshly prepared 1 % solution of sodium nitroprusside, and then a few drops of 6E.AmOH. Observe if any red colour is present after fifteen minutes and two hours. (3) Cazeneuve's test, as modified by Millard and Stark (*B.P. Conf.*, 1890, 396). To 5 ml. of the spirit add 1 ml. N/10 KMnO_4 , and observe if after two hours there is a brown turbidity or precipitate.

Any of the tests applied to a distillate from whisky will probably detect 10 % of industrial methylated spirit, and Ashby's test may show it without distillation. The presence of 10 % of mineralised methylated spirit will be shown by the milkiness of the distillate. The milkiness, however, somewhat masks the colour in the first two tests, but the addition will probably be detected by Cazeneuve's test.

Methods for the determination of methyl alcohol in the presence of ethyl alcohol have been given by Thorpe and Holmes (*Proc. Chem. Soc.*, 1904, **85**, 1), Simmonds (*S.P.A.*, 1912, **37**, 16), G. C. Jones (*S.P.A.*, 1915, **40**, 216), and also by the American Official Agricultural Chemists' Association, for proportions under 5 % (*Q.J.P.*, 1929, 424).

It has been recommended that the 1932 B.P. shall in some cases permit the use of industrial methylated spirit, subject to the Statutory Regulations concerning it.

Iso-propyl Alcohol. In 1927 a regulation was made requiring the registration of manufacturers of this alcohol, and also periodical returns to be made by manufacturers, sellers and users of it. Macdonald and Peck (*Lancet*, 1928, 443) recommended a 1.25 % solution of iodine in iso-propyl alcohol as a cheap substitute for tincture of iodine. Reif (*S.P.A.*, 1928, **53**, 497; *Analyst*, 1931, **56**, 115) and Adams and Nicholls (*S.P.A.*, 1929, **54**, 2) have given methods for its detection and determination in tinctures, etc. Walmsley (*P.J.*, 1930, Nov. 29) has given a routine test.

CHAPTER XXV

BEER. SPIRITS

Beer, ale, original gravity, names, preservatives, deleterious constituents, adulteration, prosecutions, Stout, porter. Herb, botanic beer. Spirits, adulteration, evaporation, solid extract, silent spirit, definitions, notices of dilution. Spirit of wine. Brandy, British brandy. Whisky. Rum, rum and coffee. Gin.

BEER AND ALE

BEER is an article the composition of which has varied considerably in the past. From 1802 to 1847 only malt, hops and water could legally be used. In the latter year sugar was allowed to be used ; in 1862 hop substitutes and in 1865 solid glucose were permitted. In 1880 brewers were allowed to use any saccharine matter that was not deleterious, and in 1885 the term " beer " was extended to include " any liquor which is made or sold as a description of beer or a substitute for beer, and which on analysis shall be found to contain more than 2 % of proof spirit." This definition was repeated in the Finance Act, 1910, but limited to Part II. of that Act. The Finance Act, 1914, excluded from the definition of beer any liquor not made on licensed premises having an original gravity not exceeding 1016°, and not containing more than 2 % proof spirit.

The 1885 Act prohibited under heavy penalties the addition, by brewers or retailers, of anything except finings. This prohibition is only intended to prevent the sale of water, as beer, upon which no tax has been paid. Obviously if the volume of beer, upon which duty has been paid, is increased by addition of water, the Revenue is defrauded. The enforcement of this law is only possible by the power which Inland Revenue officers have of tracing a particular beer and taking other samples for comparison.

Although there is considerable difference in the alcoholic strength and price of various beers, there is no standard quality, and as long as a publican does not add water, he can sell a cheap " beer " as dearer " ale " without any fear of punishment for adulteration. The remark made in the L.G.B. Report of 1887 is still true :—" In the present state of the law as regards the constituents of beer, it is difficult for an analyst to report that a given sample of beer is adulterated, unless it has been sold as beer of a particular brand, and thus affording the means of comparison."

Much information on brewing is given in the Report and Minutes of Evidence of the Departmental Committee on Beer Materials, 1899.

Original Gravity. The war-time "Beer (Prices and Description)" Orders of 1917, and subsequent years, provided scales of original gravity and corresponding prices, and the writer regrets that this protection of the public against overcharge has been withdrawn.

The following table gives particulars of the average composition of Birmingham samples of beer and ale analysed while an Order was in force :—

PRICE AND QUALITY OF ALE AND BEER, 1920-1

Price per pint	. . .	5d.	6d.	7d.	8d.	9d.
Gravity	. . .	1004°	1006°	1009°	1012°	1012°
Extract gravity	. . .	1009°	1012°	1015°	1018°	1019°
Original gravity	. . .	1031°	1037°	1042°	1049°	1054°
Proof spirit, v/v	. . .	6.0	6.8	7.5	8.4	9.4
Solid extract, w/v	. . .	2.2	2.9	3.7	4.5	4.8
No. of samples	. . .	38	14	46	7	4

It will be seen that with increase of price the proof spirit increases from 6.0-9.4 v/v. For revenue purposes the proportion of proof spirit present at a particular time is not satisfactory, as the alcoholic strength of a sample may be increased by fermentation, or decreased by souring. For example, a sample of bitter beer was found to contain 10.0 v/v. of proof spirit, and four days later 10.4 v/v. The original gravity, however, which relates to potential strength, remained unaltered, being 1052°. In these calculations the "gravity" of water is taken as 1000, and "original gravity" is the sum of the gravity of the unfermented extract, and a figure which is proportional to the amount of spirit present, and which represents the quantity of solid matter lost by fermentation. These two figures must be taken at the same time; and if more alcohol is formed, a corresponding amount of extract gravity is lost, and the sum of the two figures remains constant.

In 1914, after an elaborate investigation by Thorpe and Brown (*J. Inst. Brewing*, 1914, 569, 606; *Analyst*, 1915, **40**, 121, 124), a new table for calculating the relation between alcohol and loss was made legal by the Finance Act. This table has been worked out in great detail by Jones and Baker ("Original Gravity Tables"). In the absence of these tables, a close approximation may be made, if the proof spirit does not exceed about 7 v/v by the formula :—

Original gravity = Extract gravity + $(3.64 \times \text{v/v proof spirit})$.

The following example shows both methods of working. Sp. gr. of distillate 0.9945, corresponding to 6.6 v/v proof spirit. "Spirit indication" = $1000 - 994.5 = 5.5^\circ$, which, from the table, indicates 24.1° of gravity lost. By calculation, $6.6 \times 3.64 = 24.0^\circ$. By adding the extract gravity (8.0) to each figure, the original gravity is shown to be 32.1° or 32.0° .

If a beer be notably sour, the above calculation will not include original solid matter which has become acetic acid, and a corresponding amount must be added to the original gravity, as follows :—

Addition for acidity = 1·3 (acidity calculated as w/v acetic acid — 0·13.

The volatile acidity of beer is best determined by steam distillation, but titration of the alcoholic distillate will give an indication if there is any serious excess over 0·1 w/v, which is allowed for in the above calculation. Filtration of the beer in such samples is advisable.

Names. For revenue purposes the term “ beer ” included such dissimilar drinks as ale, porter, spruce beer, black beer, and any other description of beer ” (Finance Act, 1910, sect. 52). In Birmingham “ beer ” is sold at about 4*d.* per pint, “ ale ” about 6*d.*, and “ bitter beer ” about 8*d.*, but, as before indicated, there is no standard, and any attempt to establish one would be complicated by the fact that in some parts of the country “ ale ” indicates the cheapest drink.

Preservatives. From 1872 to 1874 the addition of common salt to beer was prohibited. This prohibition raised a problem for the Excise, as certain well waters and sugars were rich in chlorides, which could hardly be included in the prohibited “ common salt.” After investigation it was decided that Excise officers need not enquire into the origin of the chlorides in beer if they did not exceed the equivalent of 50 grains per gallon. In later years some analysts used this figure as a limit under the Food and Drugs Act. The Select Committee on Food Products Adulteration of 1895 reported “ The amount of salt permissible in beer urgently requires settling.”

CHLORIDES EXPRESSED AS GRAINS OF SALT PER GALLON, 1921–9.
(400 samples.)

	6–	30–	50–	70–100	Total.
Beer	35	54	11	0	100
Bitter beer	23	49	26	2	100
Ale	13	47	38	2	100

In each case about half the samples contained 30–50 grains, but the proportion over 50 grains was greater in bitter beer than in beer, and still greater in ale. The figures indicate that there is nothing unreasonable in expecting the chlorides to be less than 70 grains per gallon, a limit which the writer has used for thirty years. The figures for recent years are a great improvement on those of 1877, when 15 % of the samples contained high chlorides, resulting in vendors being fined.

In some prosecutions it has been suggested that the chlorides

will produce thirst and be prejudicial to health ; but, considering that milk contains about 100 grains of chlorides per gallon, that an adult may take 200 or more grains of salt daily in his food, and that a doctor drank water with 150 grains of salt per gallon without experiencing thirst, it is difficult to believe that claim is valid. Burgess (*S.P.A.*, 1902, **27**, 218) stated that, in water, he could just detect the taste of salt when about 25 grains per gallon were present, and that 50–66 grains gave a distinctly brackish taste.

That salt is added to produce thirst is improbable, more likely explanations being ignorance or the use of materials containing high chlorides. In one case a Birmingham publican brought a brick of salt to the Committee, when he was cautioned, and said that he only used that amount in his brew. The brick accounted for 60 grains of salt per gallon of beer ! In another case the brewer supplied samples of his brewing materials with a statement of the amount used. The total chlorides in his beer, expressed as salt, were 130 grains per gallon. The water, malt, hops and clarifier yielded 10 grains of chlorides, the glucose 29 grains, the Burtoniser 47 grains, and the salt added as such 44 grains. The Burtoniser was used to make the water harder and more suitable for brewing. It and the glucose contained unnecessarily large proportions of chlorides.

The ash of beer varies chiefly according to the amount of chlorides present ; it is usually 0.15–0.25 w/v, while the nitrogen is about 0.03 w/v.

The Preservatives Regulations of 1925, while prohibiting the use of boric and salicylic acids in “ beer,” permit the presence of sulphur dioxide up to 70 parts per million. There is no mention of ale, porter, etc., in the Regulations, so presumably “ beer ” is meant in the Excise sense to cover these drinks.

Of the samples of beer and ale bought in Birmingham 1927–9, 82 % contained 0–19 parts of sulphur dioxide per million, 13 % from 20–49, and 5 % from 50–74 parts, none of them appreciably exceeding the limit.

Experiments by Baker and Day (*J. Inst. Brewing*, 1911, 465 ; *Analyst*, 1911, **36**, 495) showed that the maximum beneficial effects on beer occurred with about 50 parts of sulphur dioxide per million. “ N.C.B.,” a writer in the *Brewers’ Journal* (1927, 436 ; *Analyst*, 1927, **52**, 717), considered that two-thirds of the permitted quantity of sulphur dioxide was sufficient, and stated that it does not disappear to any appreciable extent from a full vessel of beer.

The use of boric or salicylic acid in Birmingham beer and ale is very unusual. Of about 1,500 samples examined 1901–27, only four contained boric acid, and twenty-five salicylic acid, the maximum quantities of each being about 7 grains per gallon. A few breweries were each responsible for several adulterated samples. In one case salicylic acid tablets were being added to the beer.

Deleterious Constituents. The Licensing Act of 1872 prohibited the addition to beer of *Cocculus indicus*, copperas, tobacco (all of which had been found by the Excise chemists), and any other deleterious ingredient. Other adulterants found by them about this time were grains of paradise, ginger, cayenne, mustard, coriander and caraway seeds, and sweet flag.

Arsenic is by far the most serious impurity found in beer; particulars have been previously given (p. 79). Fortunately for Birmingham, none of the arsenical glucose came into the city, and no samples of ale or beer have been condemned for that impurity.

Another possible impurity is lead, particularly when beer has been left in pipes containing lead overnight. An inquest was held in Greenwich, in which early-morning beer had caused death by lead poisoning (*P.J.*, 1913, Aug. 30). In Middlesex beer stored in iron tanks lined with a white enamel containing lead caused a considerable number of cases of lead poisoning. The beer contained about 1 grain of lead per gallon (*B.F.J.*, 1922, 95).

Adulteration. The percentages of samples of beer reported adulterated in England and Wales are given below:—

Period	1877–	1884–	1894–	1904–13	1919–30
Percentage of adulteration .	5.3	4.2	5.9	3.8	3.4

PROSECUTIONS FOR BEER AND ALE. *Birmingham.* Salt 147.6 grains per gallon. Fine £5 (*Analyst*, 1877, 1, 215).

Stafford. Excise prosecution for having grains of paradise and adulterating beer with it. Fine £50 (*Analyst*, 1878, 2, 149).

Boston. Salt 60 grains per gallon. The chemist for the defence found chlorine equivalent to 54 grains per gallon in the beer, and 30 grains in the water from which it was brewed. He considered that the concentration of the water in brewing and the other ingredients of the beer would increase the salt in the beer by 18–30 grains per gallon. Case dismissed (*Analyst*, 1884, 9, 72).

Merthyr. Chlorine $45\frac{1}{2}$ grains per gallon, equivalent to 75 grains of common salt. Medical evidence was given for the prosecution that such an amount of salt would produce a desire for more liquid. An average normal man took 150 to 170 grains of salt per day in one form or another. Chloride of potassium would be more injurious than salt. An analyst for the defence found a similar amount of chlorine, and stated that it was made up of 63 grains of salt and 17 grains of potassium chloride; he admitted that 56 grains of chlorides had been added. A medical witness stated that he had drunk water containing 150 grains of salt per gallon without experiencing any thirst. The Bench dismissed the case, and, on appeal, the High Court (*Thorney v. Shute*, 1893) remitted the case to the magistrates to decide if the beer was of the nature, substance

and quality of the article demanded. The magistrates subsequently found that the salt used was not injurious to health, that it was added to the beer for the preparation thereof as an article of commerce, and not fraudulently to increase the bulk, weight or measure, or to conceal its inferior quality, and that it is not for the justices to determine whether the quantity was excessive. The article was therefore of the nature, substance and quality demanded (*F. & S.*, 1892, Oct. 8 ; 1983, April 22, May 13).

Birmingham. Sodium chloride 85 grains per gallon, and potassium chloride 50 grains per gallon. The brewer admitted adding salt. The Bench found: (1) The beer was not of the nature, substance and quality demanded. (2) The excess of salt was sufficient to increase thirst. (3) The excess of salt was not necessary for the manufacture of the beer. Fine £2 (*F. & S.*, 1898, Feb. 5).

West Ham. Excise prosecution for 6 gallons of water and $\frac{3}{4}$ lb. sugar per barrel. Fine £25 and costs (*B.F.J.*, 1900, 374).

Retford. Arsenic 1 grain per gallon. Brewer fined £20 for mixing, and eight directors were fined £10 each for permitting to mix arsenic, the total fines for the sample being £100 (*L.G.B. Report*, 1900 ; *B.F.J.*, 1901, 103).

Manchester. Arsenic $\frac{1}{8}$ grain per gallon, to the prejudice of the purchaser. The defence argued that the prosecution should have been for selling an article injurious to health, but the stipendiary ruled that such a prosecution would have failed as there was no guilty knowledge. He fined the defendant £2. An appeal to the High Court (*Goulder v. Rook*) was dismissed. Lord Alverstone added that it was for the magistrates to decide if the accidental introduction of a deleterious material made the article not of the nature, substance and quality demanded (*B.F.J.*, 1901, 69, 103, 196).

Birmingham. Alkaline chlorides 105 grains per gallon, being at least 50 grains in excess of the proper quantity. (40 grains were sodium chloride and 65 grains potassium chloride.) Fine £5 and costs (*Birmingham Report*, 1901).

The appeal cases *Lee v. Bent* and *Palmer v. Noblett* decided that neither a certificate "contains arsenic," nor one stating "a serious quantity of arsenic," was sufficient evidence to enable magistrates to come to a conclusion (*B.F.J.*, 1901, 196).

Manchester. Arsenic $\frac{1}{4}$ to $\frac{1}{30}$ grain per gallon. Seventeen vendors were each fined £2 and costs. The prosecutions against eighteen others, who had sold beer containing less than $\frac{1}{50}$ grain arsenic per gallon, were withdrawn (*B.F.J.*, 1901, 212).

Oxford. Excise prosecution for saccharin $\frac{1}{2}$, $1\frac{1}{4}$, and 2 grains per gallon. Fine £12 10s. on each of the three summonses (*B.F.J.*, 1901, 303).

Woolwich. Lead 0.28, 0.07 and 0.07 grain per gallon, respectively. The beer had been taken early in the morning, and some would have been standing in the pipes all night. There had been some carelessness in cleaning the pipes between the barrels and the taps. The Medical Officer of Health said there was a danger to health even in these small quantities. Fine £1 in the first case ; the other two were withdrawn (*B.F.J.*, 1912, 106).

Stoke-on-Trent. Arsenic $\frac{1}{50}$ grain per gallon. The brewery company, who admitted responsibility, was fined £15 (*B.F.J.*, 1914, 6).

Liverpool. Original gravity only 1027.7°, being nearly 3° less than it should have been according to the Beer (Prices and Description) Order. As the beer was of the correct original gravity when it left the brewery, 2½ gallons of water must have been added to the barrel. Fine £10 (*B.F.J.*, 1918, 61).

Cirencester. Sulphur dioxide 269 and 430 parts per million. Fine £10 and £12 ls. costs (*B.F.J.*, 1930, 4).

PROSECUTION FOR LAGER BEER. *Burton.* Salicylic acid 14 grains per gallon. There were three prosecutions and the vendor was fined £7 (*F. & S.*, 1894, Feb. 10).

STOUT AND PORTER

Three samples of stout bought in Birmingham gave these analytical figures :—

ANALYSES OF STOUT

	"Nourishing."	"Extra."	"London."
Proof spirit, v/v.	9.0	8.6	8.2
Solid extract, w/v.	3.6	5.0	4.6
Nitrogen, w/v.	0.052	0.078	0.065
Ash, w/v.	0.28	0.26	0.18
Chlorides, expressed as grains of salt per gallon	54	25	23

EXCISE PROSECUTIONS FOR STOUT. *Penge.* Fine of 63s. for adding sugar (*B.F.J.*, 1900, 374).

West Ham. Water 3.7 gallons per 36 gallons. Fine £50 (*B.F.J.*, 1901, 140).

PROSECUTION FOR MILK STOUT. *London, Tower Bridge.* False trade description under the Merchandise Marks Act for describing as "milk stout" an article which did not contain an appreciable quantity of lactose. Another maker used 9 lb. lactose per 36 gallons. Evidence was given that the sample of "milk stout" contained 0.41 % of proteins, while various makes of "stout" contained 0.41–0.85 %. Fine £5 (*Grocer*, 1912, Feb. 3 ; *B.F.J.*, 1912, 9, 29).

EXCISE PROSECUTIONS FOR PORTER. *West Ham.* Water 2½ gallons per 36 gallons. Fine £25 (*B.F.J.*, 1900, 374).

Thames. Water 6·2 gallons per 36 gallons. The defendant said that he had added 4 gallons of waste. Fine £30 (*B.F.J.*, 1901, 68).

HERB BEER, BOTANIC BEER, ETC.

These beverages must not contain more than 2 % of proof spirit, or they become "beer" and cannot be sold without a licence. Any fermented liquid contains alcohol, and if too much sugar is used, or if the fermentation be allowed to proceed too far, the limit may be passed, and the liquid become as intoxicating as beer.

According to Simmonds ("Alcohol, its Production, etc."), the proportion of sugar used varies from about 3–12 oz. per gallon. He gives original gravity 1012, and proof spirit 1·5 %, as a fair average. He states that saccharin is sometimes added. With carelessness as to the composition of the pipes used for conveying these drinks, lead in serious quantities may be present.

PROSECUTIONS FOR HERB BEER. *Nottingham.* Excise prosecution for 9·8 %, 5·5 % and 6 % of proof spirit, respectively, sold without a licence. Each defendant was fined 2s. 6d. (*B.F.J.*, 1900, 298).

Liverpool. Lead 4·16 grains per gallon. The drink was half barm-beer and half sarsaparilla. The lead was traced to the pipe through which the former had been drawn. Fine £5 (*B.F.J.*, 1924, 36).

PROSECUTION FOR DANDELION BEER. *Liverpool.* Lead 3·2 grains per gallon. The defendant thought the tin-washed pipes of his pump were tin-lined. It was stated they should have been block tin. Fine £1 and costs (*B.F.J.*, 1911, 55).

SPIRITS

An interesting account of the history of spirits, with illustrations, has been given by Fairley (*S.P.A.*, 1905, 30, 293).

The 1875 Food and Drugs Act gave no standards for spirits, and the standards set up varied. An appeal case (*Pashler v. Stevenitt*, 1877) held that commercial gin should not be weaker than 20° U.P. The 1879 Act fixed 25° U.P. as the limit for brandy, whisky and rum, and 35° U.P. for gin. After war-time alterations, the Licensing Act of 1921 fixed the minimum limit for these spirits at 35° U.P. and that limit was re-enacted in the 1928 Food and Drugs (Adulteration) Act, sect. 2 (2) (*d*).

Adulteration. The following table gives a tabulation of the alcoholic strength of Birmingham spirits during 1923–9, with the figures for gin bought 1874–8, for comparison :—

STRENGTH OF SPIRITS

Degrees Under proof.	Gin. 1874-8.	Gin. 1923-9.	Whisky. 1923-9.	Rum. 1923-9.
11.4°—	17	—	—	—
20.1°—	14	—	—	—
25.1°—	6	14	14	28
30.1°—	6	86	77	69
35.1°—	35	—	7	3
40.1°—	14	—	2	—
45.1°-52°	8	—	—	—
	100	100	100	100

The contrast in the figures for the two periods for gin is remarkable. No less than 57 % of the earlier samples were weaker than 35° U.P., while none of the later period exceeded that figure. Whisky was less satisfactory, 9 % of the samples being adulterated ; rum had 3 % of adulteration. The “ breaking down ” of spirits is not difficult, but profitable “ accidents ” ! are at times detected. With the present high price of spirits the addition of small amounts of water in excess may mean an appreciable increase in profits. A diminution in the adulteration of spirits has probably resulted from the increase of tied houses, as sometimes the spirits are broken down by the brewer before sending to the publican, and later on tested by the brewer's inspectors.

PERCENTAGE OF ADULTERATION OF SPIRITS, 1874-1930

Period	1874-8	1879-	1889-	1899-	1909-	1919-30
Birmingham	61.5	11.3	15.1	13.6	15.0	4.1
England and Wales	—	22.3	16.9	11.6	9.9	12.2

In the decade 1909-18, there were fifteen prosecutions for adulterated spirits in Birmingham, and £54 was paid in fines ; this fact may afford some explanation of the fall of adulteration in the next decade by 9.5 %. Not only has there been a fall in the proportion of adulteration, but also a decided decrease in the amount of water added. The average percentage of excess water in the first ten adulterated samples in 1881-6 was 16.5 %, while the last ten adulterated samples in 1923-9 only averaged 4 % of excess water. The figures for England and Wales, however, show an increase in adulteration in the last decade over the two previous ones.

In 1906 the percentage of adulteration of individual spirits for England and Wales was first tabulated, the average results for two periods are given below :—

ADULTERATION OF INDIVIDUAL SPIRITS

Percentage of Adulteration.	Brandy.	Whisky.	Rum.	Gin
1906-13	8.6	10.8	10.2	8.2
1920-30	5.2	13.5	13.8	12.9

Comparison of these figures shows that brandy only has improved, and that gin had the largest increase of adulteration.

Evaporation. Sometimes when a publican has been prosecuted he has stated that the excess water was due to evaporation of spirit, and not to addition of water. Experiments have been made in varying conditions to ascertain what justification there might be for such a defence.

In one experiment $\frac{1}{2}$ pint of whisky was put in a half-pint drinking glass and left *uncovered* for six hours. The difference in strength equalled a loss of 1.1° proof spirit, falling from 31.2° U.P. to 32.3° . The air temperature varied $60\text{--}70^\circ$ F. The next morning the spirit was just below the limit, having lost 4.1° proof spirit in twenty-four hours.

In another experiment $\frac{3}{4}$ pint of average Scotch whisky was put in a wine bottle and left *uncorked* for nine months :—

EVAPORATION OF WHISKY

Date of Analysis.	March 22.	March 29.	April 26.	May 17.	June 14.	Sept. 3.	Jan. 1.
Exposure in weeks .	0	1	5	8	12	24	40
Degrees under proof .	31.4	31.8	33.8	34.3	35.9	40.7	47.9
Excess water, % .	—	—	—	—	1.4	8.8	19.8

It will be noticed that it was not until the bottle had been left uncorked for twelve weeks that there was any indication of excess of water, and then only 1.4 %. The rate of loss was fairly uniform, being about 0.4° per week. Evaporation had reduced the volume of the sample by 2 oz.

Similar experiments have been made by Briant and Harman (*Analyst*, 1920, **45**, 448), Lowe (*S.P.A.*, 1924, **49**, 135) and McCrea and Hawken (*S.P.A.*, 1925, **50**, 66). It is evident that gross carelessness in keeping the spirit is necessary to produce an appreciable loss of strength by evaporation.

Other experiments were made by keeping spirits in *corked* bottles for years. A whisky kept in a 4 oz. bottle a quarter full lost 0.8° in eight years. Three samples of rum in the same time and conditions lost 0.3° , 0.7° and 6.3° , respectively. These spirits were $31\text{--}38^\circ$ U.P. Three samples of brandy, $15\text{--}22^\circ$ U.P., kept for seven years in full 4 oz. bottles lost $2.4\text{--}2.5^\circ$. Two samples of brandy kept twenty years lost 3.8° and 6.2° , respectively; the corking of these bottles was not very good. A sample of brandy kept twenty-five years in a bottle with the cork sealed only lost 2.7° . These three samples were $18\text{--}21^\circ$ U.P. A bottle of spirit of wine kept half full for over four years became reduced in strength from 59° to 57° O.P. It is evident that the loss of spirit strength by evaporation from corked bottles is trivial, and can very rarely be justly pleaded as a reason for excess water.

The Government chemists periodically examined four casks of spirits kept in bond. The spirit strength of three of them fell in six months from about 20° O.P. to about 10° O.P., and after two years

they were about 9° O.P. In two years the fourth cask only fell from 11.5° to 9.7° O.P. (Whisky Commission Report, ii., 232). According to a correspondent of the *Revenue Review*, casks of unfortified unsweetened juices, about 70° U.P., lose little alcohol during a storage of about four years. Five casks lost nothing, and four others 0.3–0.6° (*P.J.*, 1905, Oct. 28).

Solid Extract. The amount in spirits is usually small; the tabulation below gives the amounts found in Birmingham spirits, 1922–9 :—

Solid extract, w/v	0-0.1	0.1-0.2	0.2-0.3	0.3-0.4	0.4-0.5	0.5-0.6	0.6-0.7	0.7-0.81	Total.
Gin	100	—	—	—	—	—	—	—	100
Irish whisky	8	80	6	3	—	—	3	—	100
Scotch whisky	10	74	12	2	2	—	—	—	100
Rum	—	—	4	48	15	11	15	7	100

It is evident from the above figures that with gin and some whiskies the sp. gr. without distillation will give almost the correct alcoholic strength, as the solid extract is so small. Rum, on the other hand, will require correction for the “obscuration,” as has been previously explained (p. 351). One Birmingham sample of whisky had 1.0 w/v of solid extract, owing to the presence of sherry. W. Partridge has given particulars of the amount of extract found in brandy (*S.P.A.*, 1929, **54**, 154) and whisky (*S.P.A.*, 1931, **56**, 177).

Cayenne has sometimes been added to spirits so that its pungency may increase the apparent strength of the spirit. Kellermann (*Analyst*, 1898, **23**, 209) found that the residue after distillation of such a sample had a lasting burning sensation when applied to the lips.

Silent Spirit. For all practical purposes the effects of spirits for good or evil are proportional to the amount of alcohol present; the characteristic differences between them are due to “secondary products,” which rarely exceed 0.4 w/v of the spirit. The amount present depends somewhat on the materials distilled, but more on the nature of the still used. The older form is the “pot still,” which yields spirits containing a larger proportion of secondary products than the “patent still,” which was invented by Coffey in 1831. The former is worked intermittently and the latter continuously, and by it the purity and strength of the spirit can be varied at will. Spirit of wine, also called “silent” or “neutral” spirit, is a strong spirit prepared by the Coffey still, and is free from secondary products. The difference may be illustrated by the following averages of analyses of samples bought in Birmingham about 1905. “British brandy” is an imitation of brandy largely composed of silent spirit; the mixtures were intended to be sold as genuine brandy :—

SECONDARY PRODUCTS IN SPIRITS

	Brandy.	British Brandy.	Mixtures.	Spirit of Wine.
Number of samples . . .	14	4	2	1
Volatile acidity, as acetic acid .	82	14	50	3
Fixed acidity, as tartaric acid .	24	3	9	0
Aldehyde	13	3	5	3
Furfural	1·7	0·2	0·7	0
Ethers, as ethyl acetate .	111	4	—	0
Higher alcohols (Vasey test) .	790	40	50	0
Solid extract, w/v . . .	0·8	0·4	0·4	0

With the exception of the solid extract, the figures represent gm. of secondary products per 100,000 of absolute alcohol. The expression of them in this form is better than on the brandy itself, as it avoids the use of decimal places, and compares them on a water-free basis. The addition of either water or silent spirit reduces the proportion of secondary products.

In 1903–5 there were a number of prosecutions for selling brandy adulterated with silent spirit, and in the latter year two publicans were fined for selling pot still whisky as “Scotch whisky” and “Irish whisky,” respectively. An appeal to Quarter Sessions was inconclusive, and a Royal Commission on Whisky and Other Potable Spirits was appointed to enquire into the question. A Final Report was issued in 1909. It and two volumes of Minutes of Evidence give a large amount of information on the subject.

Owing to the present high prices of spirits, the samples of them submitted to public analysts are usually too small to permit the tests for silent spirit to be made. For a formal sample one-third of a quart of spirits would not be too much. In recent years there have been few prosecutions for adulteration with silent spirit.

For methods of analysis reference may be made to Vasey's “Analysis of Potable Spirits,” 1904; Simmonds' “Alcohol, its Production, etc.,” 1919; to the Whisky Commission Reports; and to the papers of Allen and Chattaway (*S.P.A.*, 1891, **16**, 102), of Schidrowitz and Kaye (*S.P.A.*, 1905, **30**, 149, 190; 1906, **31**, 181), of Hehner (*S.P.A.*, 1905, **30**, 36), of Veley (*J.S.C.I.*, 1906, 398), of Mann with Stacy or Kirton (*J.S.C.I.*, 1906, 1125; 1907, 450), and of Bedford and Jenks (*J.S.C.I.*, 1907, 123).

Definitions. The conclusion of the Whisky Commission was, “The term ‘brandy’ is applicable to a potable spirit manufactured from fermented grape juice and from no other materials,” irrespective of the “apparatus or process used in distillation.” A section of the Spirits Act, 1860, which was repealed in 1880, stated: “All spirits which shall have had any flavour communicated thereto, and all liquors whatsoever which shall be mingled or mixed with any such spirits, shall be deemed a British compound called ‘British brandy.’” The Commission considered such a spirit was entitled to be sold as “British brandy.”

Whisky, according to the Commission, is "a spirit obtained by distillation from a mash of cereal grains (including maize) saccharified by the diastase of malt," and that "Scotch whisky" and "Irish whisky" are whisky as above defined distilled in Scotland and Ireland, respectively. This definition includes the products of both pot and Coffey stills, and blends of them.

The L.G.B. Reports of 1888 and 1891 mention adulteration of whisky with tincture of guaiacum and treacle. A minute trace of copper is frequently found in pot still spirit (Whisky Commission Report).

There are two varieties of rum. Jamaica rum is prepared only by the pot still, and contains a larger amount of secondary products than Demerara rum, some of which is made in Coffey stills. The Commission considered that either type might be sold as "rum." They accepted the definition "Rum is a spirit distilled direct from sugar-cane products in sugar-cane growing countries."

Williams has published analyses of Jamaica rum (*J.S.C.I.*, 1907, 498). Micko (*Analyst*, 1909, **34**, 54; 1910, **35**, 208) has suggested a test for discriminating between Jamaica rum and artificial rum. The L.G.B. Report for 1912 mentions a rum containing 9.9 % of glycerin.

A definition of gin given to the Commission was, "Gin is made of spirit specially rectified and re-distilled with juniper berries and other flavouring herbs."

NOTICES OF DILUTION OF SPIRITS. References to High Court decisions are given below :—*Sandys v. Small*, 1878 (whisky), *Gage v. Elsey*, 1883 (gin), *Morris v. Johnson*, 1890 (whisky), *Morris v. Askew*, 1893 (rum), *Palmer v. Tyler*, 1897 (rum), *Preston v. Grant*, 1924 (whisky), *Rodbourn v. Hudson*, 1924 (rum).

PROSECUTION FOR SPIRITS OF WINE. *Doncaster*. Very deficient in alcohol. Fine 10s. (*B.F.J.*, 1900, 264).

PROSECUTIONS FOR BRANDY. *Yorkshire, East Riding*. "Reduced from 25° under proof to 27.6° under proof." The magistrates dismissed the case, considering that the certificate was not in proper form, but on appeal (*Findley v. Haas*) the High Court held it was sufficient and remitted the case with directions to convict (*B.F.J.*, 1903, 87).

Hamilton. "At least 65 % of the spirit was not derived from grapes." "Genuine brandy contains at least 42 parts of ethers and 0.85 parts of furfural per 100,000 parts of brandy." The analysis showed 13.25 parts of ethers (calculated as ethyl acetate), and 0.35 parts of furfural per 100,000 of brandy. It was 22.5° U.P. For the defence it was maintained that practically all commercial brandy in this country was made from grain spirit. The Sheriff inflicted a nominal penalty of £1 in each case. An appeal to the Scotch High Court followed (*Wilson and McPhee v. Wilson*) and was

dismissed (*B.F.J.*, 1903, 165, 181; Whisky Commission Final Report, p. 33).

North London. Spirit not derived from the juice of the grapes at least 60 %. The bottle was labelled "Fine old pale brandy." The Public Analyst found 40.9 parts of ethers per 100,000 parts of alcohol, and an analyst for the defence 32 parts, and also 468 parts of higher alcohols. After five days' hearing the learned magistrate suggested a standard of 80 parts of ethers, decided that the sample was not brandy, and fined the defendant £5 and £50 costs (*B.F.J.*, 1904, 87, 134; *P.J.*, 1904, May 7, 14, 21, June 4).

Preston. "An imitation of brandy, often called 'British Brandy'." Fine £2 (*B.F.J.*, 1904, 116).

Kensington. 58 % of the spirit was not derived from grape juice or wine. Fine £5. The appeal to Quarter Sessions was dismissed with costs (*B.F.J.*, 1906, 7).

Maidstone. 27.5° U.P. instead of 25°. On the label of the bottle was printed "30 u.p." in small type. The chairman of the Bench thought that everyone who bought brandy knew what the letters "u.p." meant, but they were not legible enough to protect the defendant, who was fined 42s. (*B.F.J.*, 1915, 39).

Liverpool. Spirit other than brandy, 45 %. The esters were 45 parts per 100,000 parts of absolute alcohol, instead of 80 parts. Case dismissed on proof of warranty (*B.F.J.*, 1923, 93).

PROSECUTIONS FOR BRITISH BRANDY. *West Powder.* "41° under proof, which is 16° more than is allowed by law." It is adulterated to this extent by addition of water." Fine 10s. (*F. & S.*, 1893, April 22).

Penzance. Brought down to 39° under proof. For the defence it was maintained that there was no standard for British brandy, and that if there were, it would be that for gin, 35° U.P. The magistrates ruled that a technical mistake had been made, and that the article did come within the Act. Fine 5s. (*Grocer*, 1907, Jan. 19).

PROSECUTIONS FOR WHISKY. *Derby.* Mixed with 30 % of water. There were prominent notices in the premises:—"All spirits sold here are mixed." The magistrates dismissed the case, and their decision was confirmed on appeal (*Sandys v. Small*). The High Court ruled that notice of admixture could be given to the purchasers in other ways than by a label (*Analyst*, 1878, 3, 314).

37° U.P. The sample was sold in a club-room in which there was no notice; in two other rooms there were notices "All spirits sold are diluted." The case was dismissed, but on appeal (*Morris v. Johnson*) the High Court held that the justices should have ascertained if the purchaser knew if the spirits sold were diluted, and if he did not know, they should have convicted (*Analyst*, 1890, 15, 60).

Chippenham. Added water 10.68 %, in addition to that

permitted by the Act, being 35·68° U.P. Evidence was given that the whisky had been received a year and a half previously, that the strength then was 23° U.P., and that no water had been added to it, the deficiency being due to evaporation. An Excise officer stated that 12 % was allowed by the Customs for evaporation in bond not exceeding two years. The magistrates dismissed the case, finding that no water had been added. This decision was confirmed on appeal to the High Court, *Smith v. Jeffreys* (*F. & S.*, 1897, March 13, May 29).

Hamilton. 27° U.P. The Government analysts found 25·8° U.P. and stated that as the bottle was half full and imperfectly corked, the sample might have been 25° U.P. when sold. Case dismissed (*F. & S.*, 1898, March 19).

Wolverhampton. Cayenne 3 grains per gallon. It was stated that the object of the addition was to give a fictitious strength. Fine £2 5s. (*B.F.J.*, 1899, 24).

Bolton. Whisky made from methylated spirit. The defendant, who had been previously fined for similar offences, was fined £51 (*B.F.J.*, 1899, 191).

North London. Irish whisky. 174·5 parts of secondary constituents per 100,000 of absolute alcohol. Furfural was absent. *Scotch whisky.* 110·5 parts of secondary constituents, including 0·5 of furfural, per 100,000 of absolute alcohol. The Public Analyst considered that each sample consisted entirely of patent still, silent or neutral spirit. He stated that whisky should consist of spirit distilled in a pot still, and that such spirit contained at least 380 parts of secondary constituents, while patent still spirit contained 89 to 204 parts of secondary constituents per 100,000 of absolute alcohol. For the defence it was admitted that 90 % of each sample was patent still spirit, but it was maintained that such articles were largely and legitimately sold as Irish and Scotch whisky. One sample was marked "Fine old Scotch whisky" though it was only a year old. After eight days' hearing of the case the magistrate decided that patent still spirit was neither Irish nor Scotch whisky, and that the articles had been sold to the prejudice of the purchasers, and fined each defendant £1 and £100 costs. An appeal against the conviction was heard at the Quarter Sessions, but no decision was given as the magistrates were unable to agree (*B.F.J.*, 1905, 229, 249; 1906, 17, 36, 54, 75, 116, 135; *Grocer*, 1905, Nov. 6, etc.).

North London. Alcohol 34·19 %, water 65·19 %, and non-volatile matter 0·62 %; the strength of the sample was accordingly 28·2° under proof. The magistrate dismissed the case as the certificate was bad, requiring a mathematical calculation before it became intelligible: a certificate must bear on the face of it an indication as to what was really wrong with the sample (*B.F.J.*, 1910, 40).

Kensington. 44·13 degrees under proof. The bottle was labelled "Half Proof," which the Bench considered was sufficient disclosure and dismissed the case (*B.F.J.*, 1911, 80).

Birmingham. Scotch whisky. Alcohol 25·4, brown solid matter 1·0, water, etc., 70·4—total 96·8 grammes per 100 cubic centimetres. Whisky 25° U.P. should have 34 grammes of alcohol per 100 cubic centimetres; the sample therefore contained 25 % of water in excess of 25° U.P. The defence was that the whisky had been accidentally mixed with sherry. Fine £5 (1913 Report).

Leeds. Water, more than the permissible maximum, 3 %. For the defence it was urged that the loss was entirely due to evaporation. The sample had been taken from a 28-gallon cask, only about one-third full. It was stated that in a bonded warehouse about 2 % per annum was lost by evaporation. Defendant ordered to pay costs (*Analyst*, 1922, 47, 475).

Sutton Coldfield. Excess water 11%, being 42·46° under proof. There was a notice in the bar, "All spirits sold on this establishment are diluted, and no alcoholic strength is guaranteed." The justices held the notice to be sufficient, although the purchaser had neither seen the notice, nor had his attention called to it, and dismissed the case. On appeal (*Preston v. Grant*) the case was remitted to the justices with directions to convict. It was ruled that a seller, who relied on a notice, must prove that the notice was conveyed to the purchaser, and was understood by him (*Analyst*, 1924, 49, 581).

London, Bow Street. 48° U.P. A notice was displayed having the same wording as in *Preston v. Grant*, but as the price charged was not unreasonable for the dilution, the magistrate considered the notice sufficient and dismissed the case (*Analyst*, 1925, 50, 133; *B.F.J.*, 1925, 30).

Birmingham. Whisky of 35° U.P. 95 %, excess water 5 %. This opinion was based on the fact that the sample was 38·5° U.P. Fine £2 (1926 Report).

North London. 48° U.P. There was a notice, "All spirits sold in this compartment are diluted below 35° U.P." The magistrate considered such notices were for evading the Act, and were played out. Fine £5 (*B.F.J.*, 1926, 19).

North London. 37·9° U.P. As the non-volatile matter was 2·66 % instead of 0·2–0·3 %, the inspector considered the sample had been doctored. Fine £5 (*B.F.J.*, 1927, 100).

PROSECUTIONS FOR RUM. *Stafford.* Diluted 19 % beyond the statutory allowance of 25 %. The magistrates dismissed the case, holding that the exhibited notice, "All spirits sold at this establishment are diluted according to the new Excise regulations," was sufficient. On appeal, the High Court held (*Morris v. Askew*) that the mere notice itself was not a protection, and that the

magistrates should have determined whether the purchaser was prejudiced (*F. & S.*, 1893, Nov. 18).

"Excess of water above what is allowed by Act of Parliament, and I estimate the excess is 13 % of the sample." The magistrates dismissed the case, being of opinion that an "estimate" was not sufficient to justify a conviction. On appeal (*Newby v. Sims*), the High Court saw no objection to the word "estimate," but found the certificate bad, not showing how the "excess" was calculated. "Act of Parliament" did not necessarily mean the 1879 Act (*F. & S.*, 1894, Jan. 27).

Dartford. "27.4° U.P., 25 % under the legal limit." The case was dismissed as the certificate did not specifically state that the rum was adulterated with water (*F. & S.*, 1894, June 9).

Brentford. Proof spirit 70.6 and water 29.4. There was a notice in the bar, and the purchaser had knowledge of the dilution. The magistrates fined the vendor, considering that he should have declared the dilution by a label on the bottle. On appeal (*Palmer v. Tyler*) the High Court quashed the conviction (*F. & S.*, 1897, June 12).

Wolverhampton. Cayenne 5 grains per gallon. Fine £2 5s. and costs (*B.F.J.*, 1899, 24).

Westminster. Below the statutory strength, and composed of a mixture of rum, whisky and gin. Ordered to pay 14s. 6d. costs (*B.F.J.*, 1902, 6, 20).

Stockport. Silent spirit between 70 % and 80 %. The Public Analyst stated that the sample contained only 62 parts of compound ethers per 100,000 of alcohol, while genuine Jamaica rum contained 400 to 600 parts. It was Demerara rum prepared by the Coffey still. For the defence it was stated that the article was sold as imported, and that three times as much rum was imported from Demerara as from Jamaica. The case was dismissed, as the magistrates were of opinion that the sample corresponded more or less closely with the accepted analyses of Demerara rum. The defendant was allowed 10 guineas costs (*B.F.J.*, 1904, 214).

Lanark. "Jamaica rum" deficient in esters. It was stated that the article was sold as exported from Jamaica, and the defendant was ordered to pay 38s. costs (*B.F.J.*, 1906, 32).

London, Marylebone. Three prosecutions of one vendor were instituted by the Government of Jamaica for the application of a false trade description—namely, "Jamaica rum"—to spirits., Fine £60 and 10 guineas costs (*Grocer*, 1907, April 27 ; *B.F.J.*, 1907, 85).

Hampstead. 41½° U.P. The purchaser saw and read a notice in the bar: "All spirits sold in this establishment are of the same superior quality as heretofore, but, as required by the Food and Drugs Adulteration Act, they are now sold as diluted spirits, no

alcoholic strength guaranteed." The Bench considered the sale was to the prejudice of the purchaser, the notice being ambiguous and misleading, and did not convey to the mind of the purchaser the fact that when he asked for rum he was being supplied with spirit which was more than 35° U.P. Fine £1 and 5 guineas costs. The vendor appealed (*Rodbourn v. Hudson*), and the appeal was dismissed. "Superior quality" was considered by the High Court to be misleading when used for "diluted spirit." Further, the Sale of Food and Drugs Act contained no obligation to sell spirits as "diluted spirits," and "no alcoholic strength guaranteed" might mean that the vendor did not guarantee any particular strength over the minimum. The Court held that a purchaser must be told in substance that the thing he is getting is not the thing he asked for. If the notice, properly construed, did not convey the required information, the justices must not acquit (*B.F.J.*, 1924, 40, 105, 115; *Analyst*, 1924, 49, 229, 582).

PROSECUTION FOR RUM AND COFFEE. *Bradford*. Bought at a refreshment room. No alcohol and only a trace of rum essence. For the defence it was stated that the concentrated rum and coffee essence, used for mixing with the coffee, contained less than 2% of proof spirit, owing to evaporation of spirit during keeping. Fine £2 (*B.F.J.*, 1930, 50).

PROSECUTIONS FOR GIN. *Braintree*. 40½° U.P., being 5½° below the minimum strength. At the time of sale the vendor called the attention of the purchaser to a large notice: "All spirits are sold as diluted and no alcoholic strength is guaranteed." The vendor was fined £2 and costs. On appeal (*Gage v. Elsey*), the conviction was quashed as the vendor was protected by the notice although the strength was below the minimum of the 1879 Act (*Analyst*, 1883, 8, 850).

Retford. Added water 14 parts, gin of lowest legal strength 86 parts. The case was dismissed, the Bench holding that it was the analyst's duty simply to say how many parts of gin and water were present and to leave it to the Bench to say if it was of the lowest legal strength (*F. & S.*, 1894, June 30).

Poplar, London. Alcoholic strength deficient to the extent of 4.7 % of proof spirit. Case dismissed as the proportion of water and other percentages were not given. The defendant was allowed 21s. costs (*F. & S.*, 1895, Jan. 19).

Richmond. Water, over and above that contained in gin 35° under proof, 7¼ %. The bottle was inconspicuously marked "42 U.P." The Bench were of opinion that there was not sufficient disclosure to the purchaser, and fined the defendant £5 (*F. & S.*, 1898, Feb. 5).

CHAPTER XXVI

WINES. CORDIALS. MEDICATED WINES

Port. Sherry. Madeira. Cider. British, home-made, temperance, etc., wines. Prosecutions. Cordials, lime juice, lemon squash, ginger brandy, etc. Prosecutions. Medicated wines, quinine, ipecacuanha, beef and malt, etc. Prosecutions.

THE adulterations of wine may be divided into three classes : (1) Substitution of one kind for another. (2) Fictitious wines made by colouring and flavouring sugar solutions. (3) Addition of prohibited preservatives or excess of permitted ones.

Port and **Madeira** have the distinction of being protected by the Anglo-Portuguese Commercial Treaty of 1914, and they must be the produce of Portugal. Further, in 1916 it was enacted that port must be accompanied by a certificate that it was produced in the Douro district. There have been numerous prosecutions under the Merchandise Marks Act for substitution, and such descriptions as "Tarragona port" have been held to be false trade descriptions.

Kickton and Murdfield (*Analyst*, 1913, **38**, 368) have given a summary of numerous analyses of **port wine**. They found, alcohol 18–22 v/v, total solids 8.0–11.5 %, ash 0.15–0.3 %, glycerol 0.5–0.7 %, and phosphoric acid 0.015–0.030 %. Eight samples of port bought in Birmingham about 1890 gave the following averages : proof spirit 35.5 v/v, total solids 8.0 w/v, ash 0.21 w/v.

The name "**sherry**" is derived from Xeres, a town in Andalusia, in Spain, and vendors have been fined for selling British wines under this name.

ANALYSES OF SHERRY (twenty-five samples)

PROOF SPIRIT v/v . . .	27.4–28.8	31.6–32.3	34.3–35.8	36.7–38.9	Total.
Percentage of samples . .	12	40	32	16	100
TOTAL SOLIDS w/v . . .	3.1–	4.1–	5.0–5.7	6.2–6.4	Total.
Percentage of samples . .	32	32	28	8	100
ASH w/v	0.27–	0.40–	0.50–0.51		Total.
Percentage of samples . .	29	54	17		100
SO ₄ , CALCULATED AS K ₂ SO ₄ , w/v	0.21–	0.30–	0.40–0.41		Total.
Percentage of samples . .	20	60	20		100
ACIDITY, AS TARTARIC ACID, w/v	0.11	0.37–0.49	0.50–0.57	0.66	Total.
Percentages of samples . .	4	67	25	4	100
ROTATION IN 200 mm. TUBE + 1.6°		–0.6 to 0.9°	–1.0 to –1.3°	–2.7°	Total.
Percentage of samples . .	6	27	61	6	100

The sp. gr. of the samples varied from 0.992–1.003, and the ash exceeded the SO₄, calculated as K₂SO₄, by 0.05–0.10 w/v.

Salicylic acid was only detected in the sample containing the smallest amount of proof spirit. The 1923 Report of the Ministry of Health mentions four samples of sherry containing 0.04–0.10 % of boric acid.

Analyses of **Madeira wine** have been given by Silva (*Analyst*, 1911, **36**, 410) and by Kickton and Murdfeld (*J.S.C.I.*, 1915, 1025). Schidrowitz (*J.S.C.I.*, 1907, 629) published notes on claret, and Rosenheim and Schidrowitz (*S.P.A.*, 1900, **25**, 6) gave analyses of dry champagne. Methods for the detection of other fruit wine in grape wine have been given by Röttgen (*Analyst*, 1927, **52**, 39) and Rüdiger and Diemair (*Analyst*, 1927, **52**, 599).

Numerous analyses of apple juice and **cider** have been given by Embrey (*S.P.A.*, 1891, **16**, 41), A. H. Allen (*S.P.A.*, 1902, **27**, 183), and Barker and Russell (*S.P.A.*, 1909, **34**, 125); see also Bufton (*Analyst*, 1927, **52**, 649).

The following table gives the composition of samples of cider, including an analysis of one that was condemned as containing at least 25 % of water in excess :—

ANALYSES OF CIDER, 1915–8

	Six Samples.	Watered Sample.
Proof spirit, v/v.	7.0–9.5	3.2
Alcohol, w/v.	3.20–4.32	1.46
Solid extract, w/v.	1.2–3.8	3.5
Ash, w/v.	0.22–0.30	0.13
Total acidity, as malic acid, w/v.	0.39–0.78	0.27
Volatile acid, as acetic acid, w/v.	0.11–0.41	0.10
Original solids, w/v.	8.7–11.9	6.6
K, reducing sugar, w/v.	0.2–2.2	2.8
Sp. gr.	1.001–1.010	1.012
Rotation, 200 mm. tube	0 to –2.4	–0.8

The original solids were calculated as suggested by Allen (*opus cit.*, 191), being : alcohol w/v \times 2.07 + acetic acid \times 1.5 + solid extract. Each sample gave the reaction for apple juice described by Barker and Russell (*opus cit.*, 132). The cider, with or without concentration, was shaken for five minutes with an equal volume of ethyl acetate. After separation, the ethyl acetate extract was carefully poured on the surface of lime-water in a test tube, when a yellow colour was produced where the liquids joined. Boric acid was not detected in the samples, but one of them contained about $\frac{1}{3}$ grain per pint of salicylic acid. One sample, which was suspected of causing lead poisoning, had about 0.24 lead per 100,000.

The L.G.B. Report for 1911 refers to samples of cider taken in Blackpool; thirteen of the fourteen examined were neither fermented nor prepared from apples, but were coloured, flavoured, and aerated sugar solutions. “National Mark” cider is required to be free from concentrated fruit juices, saccharin, artificial bouquets and essences,

and to have an original gravity of not less than 1.040 at 60° F. Of the samples of cider examined in England and Wales during 1906-13, 15.9 % were reported adulterated.

British wines are included in the legal term "sweets," which is defined (Statutory Rules and Orders, 1927, No. 728) as "Any liquor which is made from fruit and sugar, or from fruit and sugar mixed with any other material, and which has undergone a process of fermentation in the manufacture thereof." If foreign wine be used for flavouring, not more than 15 parts must be mixed with 100 parts of British wine, and only in the course of manufacture, and the mixture must be sold as "British wine."

In 1924, Hancock made a detailed "Report on the Composition of Commoner British Wines and Cordials" (Ministry of Health Report on Public Health, No. 24) which included analyses made by the Government chemist on 357 samples, including essences, etc., used for making them (*Analyst*, 1924, **49**, 387). Russell and Hodgson have given analyses of eight samples of ginger wine (*S.P.A.*, 1911, **36**, 60).

Orange wine was described by the 1914 B.P. as being made by the fermentation of a saccharine solution to which fresh bitter orange peel has been added. It was required to contain 12-14 v/v of alcohol, which corresponds to 21-24.5 v/v proof spirit. The fifteen samples given in Hancock's Report varied from 18.04-26.65 v/v proof spirit, 10.66-24.57 w/v extract, and 0.17-0.59 w/v ash.

Home-made wines, such as raisin, damson, elderberry and rhubarb, have been made by the housewife by adding water to the fruit and sugar and fermenting with yeast. A home-made rhubarb wine examined by the writer contained 22 v/v proof spirit, and 20.5 w/v of extract, of which 1.0 % was ash. Sometimes the failure to recognise the alcoholic strength of such beverages leads to distressing results. Two instances have come under the writer's notice. A lay preacher invited a scholar to have some of his home-made wine; the young man liked it, and his host perceived, to his consternation, that his guest was so inebriated that he had to be seen home. In another instance, a police constable was pressed by a man who owed him a grudge to have some of his home-made wine; the hoped-for result was achieved, and the unsuspecting constable was severely reprimanded for being drunk on duty.

Some years ago "Bees wine" was largely made. A solution of sugar and treacle was fermented by the "ginger beer plant," which is a symbiotic ferment consisting of a yeast and a bacterium. One sample after three days' fermentation contained 3.5 v/v proof spirit, after ten days, 6.6 v/v, and after twenty-six days, 12.1 v/v. The solid extract fell in that time from 15.1 w/v to 7.5 w/v. Another sample contained 21.4 v/v of proof spirit (see Holmes, *P.J.*, 1920, Jan. 3).

Unfortunately, such **alcoholic wines** as orange, ginger, etc., have counterparts in non-alcoholic wines, *sold under the same names*. With such ignorance of the results of fermentation as has been indicated, it is not surprising that vendors may be careless in substituting one kind for the other. One Birmingham vendor sold a bottle labelled "Guaranteed a pure British wine manufactured from freshly gathered cowslips," and said, "I don't really know whether this is non-alcoholic, but it won't hurt you." It contained 22·4 v/v of proof spirit, and was sold in prohibited hours. In another case a Birmingham inspector asked at a large shop for non-alcoholic ginger and orange wines. The man at the counter said they were non-alcoholic, and when challenged on the statement, fetched his chief, who confirmed him. The wines had 24·2 v/v proof spirit, and 20·0 v/v, respectively. Prosecutions have resulted from such substitutions. In one instance the manufacturer of a wine labelled "Warranted free from spirit," which contained 7·48 v/v of proof spirit, explained that the label was intended to indicate that the wine was free from *added* spirit. When about 30 v/v of proof spirit is produced, fermentation ceases owing to the death of the yeast. Wines which are stronger than this, such as port, have had brandy or other spirit added.

Hancock (*opus cit.*) states that the larger proportion of the alcoholic British wines are made by flavouring a more or less characterless basis wine. It seems reasonable to expect that there should be some relation between the wine and the fruit whose name it bears. There have, however, been magisterial decisions to the contrary. The amount of solid extract in five such samples bought in Birmingham varied from 18·4–23·2 w/v.

ADULTERATION OF WINES IN ENGLAND AND WALES

Period	1877–81	1882–91	1892–1901	1902–13	1919–30
Percentage of adulteration	14·2	4·5	3·2	12·9	3·2

Non-alcoholic **temperance wines** are syrups which have been flavoured and sometimes coloured with "fast red" or other coal-tar dye. As they must not contain more than 2 v/v of proof spirit, some preservative is necessary. The Preservative Committee of 1901 recommended that the salicylic acid used in such drinks should not exceed 1 grain per pint, and that its presence should be declared. The Preservative Regulations of 1925, while prohibiting the use of any preservative in alcoholic wines, permitted the use of either 350 parts of sulphur dioxide, or 600 parts of benzoic acid, per million, in non-alcoholic wines, cordials and fruit juices, if the presence of the preservative was declared on a standard label.

Scott Dodd has recently shown (*S.P.A.*, 1929, **54**, 19) that the natural occurrence of boric acid in wines only amounts to 10–30 parts

per million, but that concentrated imported grape juice may contain an appreciable quantity.

Thirteen of the fifteen non-alcoholic wines, bought in Birmingham, contained 0.1-1.7 v/v proof spirit; the other two, 2.3 v/v and 2.6 v/v, respectively. Five samples had 21.0-25.1 w/v solid extract; five others, 27.2-30.7 w/v; and one, which should have been called a cordial, had 43.7 w/v. Five samples examined in 1929 had 230-350 parts of benzoic acid per million, and five others 85-240 parts of sulphur dioxide per million.

During 1906-13, 35.8 % of the samples of "non-alcoholic wines" examined in England and Wales were adulterated; 24.6 % of those examined 1920-7, and 3.4 % during 1928-30.

PROSECUTIONS FOR PORT WINE. *Lambeth.* False trade description, "Fine blended Tarragona Port," applied to wine which was not blended. One-third of it was Tarragona port, and two-thirds wine made in England from currants and raisins. The magistrate decided that "blended" could not be applied to mixtures of dissimilar liquids, and fined vendor £2 and 15 guineas costs (*Grocer*, 1905, Sept. 23, 30; *B.F.J.*, 1905, 207).

London, Worship Street. False trade description, "Tarragona port blended with wine produced from finest foreign grapes," applied to wine one-third of which was Tarragona port, and two-thirds a liquid made in Greece from the concentrated juice of fresh grapes. The magistrate decided that, though the word "finest" was open to question, the Act had not been infringed, and dismissed the case, allowing ten guineas costs. On appeal, the High Court held that the word "finest" was only an appreciation of the vendor, and did not make the description false (*P.J.*, 1905, Dec. 16; 1906, June 2).

Gainsborough. A large amount of sugar, a small proportion of which was fruit sugar, and was devoid of alcohol. Sp. gr. 1.049. It was labelled "Genuine unfermented British port wine." Fine £3 (*B.F.J.*, 1906, 34).

Lambeth. False trade description, "Fine British Tarragona Wine," applied to a wine which was 85 % British wine from imported raisins and 15 % wine from Tarragona. The magistrate dismissed the case as he considered the label did truly describe the contents of the bottle. On appeal (*Holmes v. Pipers*), the High Court held the label was a false one as it assumed too high a standard of knowledge in the public, who might not see anything antagonistic between the words British and Tarragona. On re-hearing, the defendant was fined £10 and 10 guineas costs (*Grocer*, 1913, Feb. 22, March 8, 22, April 12, Oct. 25, Nov. 8).

Hexham. Only 24 v/v of proof spirit and much below genuine port wine, which contains about 36 v/v of proof spirit. The case was dismissed, the Bench holding the evidence was insufficient to prove an offence (*Grocer*, 1915, Jan. 9; *B.F.J.*, 1915, 19).

Preston. False trade description, "Port," applied to an article which contained: invert sugar 25 %, tartaric acid 0.6 %, proof spirit 1 %, fruit juice and a little salicylic acid. It was also labelled "Entirely free from alcohol." The defendants explained that a small label "Flavour" should have been placed below the word "Port." Fine £10 and 50 guineas costs (*Grocer* and *P.J.*, 1922, Oct. 7).

Mortlake. Application of a false trade description, "Tarragona Port," to a cheap Spanish wine, contrary to the Merchandise Marks Act and the Anglo-Portuguese Commercial Treaties, 1914-16. The magistrates refused to convict the defendant, but on appeal (*Sandeman v. Gold*) were ordered by the High Court to do so (*Justice of the Peace*, 1924, 10; *Grocer*, 1924, Oct. 20).

PROSECUTIONS FOR SHERRY WINE. *Keighley.* Alcohol 0.26 %, water 79.70 %, extractive matter 20.04 %. Fine 5s. (*B.F.J.*, 1903, 136).

Birmingham. Applying a false trade description, "Sherry," to a non-alcoholic liquid. The bottle was also marked "Non-alcoholic." Evidence was given that the word "sherry" was derived from Xeres, a town in Andalusia, Spain. Fine £2 (*Analyst*, 1926, 51, 33).

London, Old Street. Ethyl hydroxide 14.44 per cent. by volume, corresponding to a deficiency of 9.7 per cent. The defence was that the B.P. standard of 16 per cent. by volume applied to medicinal mixtures, and that the article was sold as "British Sherry." The magistrate considered the purchaser had been given ample notice of what he was purchasing, and dismissed the case, allowing the defendant 5 guineas costs (*B.F.J.*, 1931, 70; *Grocer*, 1931, June 13).*

PROSECUTIONS FOR PORT, BURGUNDY, TARRAGONA AND MEDOC WINES. The vendor was prosecuted for applying false trade descriptions:—"Fine old port," "Red Burgundy," "Tarragona," and "Special Medoc, Bordeaux"—to basis wines which had not come from Oporto, Burgundy, Tarragona, and Medoc, respectively. Fine £10 in each case (*Grocer*, 1905, Dec. 23; *B.F.J.*, 1906, 74).

PROSECUTION FOR MOSELLE WINE. Applying a false trade description, "Berncastle Doctor," to wine which had not come from the Berncastle vineyard on the Moselle. Fine £5 (*B.F.J.*, 1908, 161).

PROSECUTION FOR CIDER. *Bristol.* Applying false trade description, "Cyder," to a liquid not containing apple juice. It was stated to contain saponin. Fine £5 (*P.J.*, 1907, Sept. 22; *B.F.J.*, 1907, 152).

Leyburn. Artificial liquid consisting of aerated water, sugar, and flavouring materials. Fine 1s. (*B.F.J.*, 1909, 164).

Dublin, Southern. No apple juice, being sugar, water and a little tartaric acid. Fine £2 (*B.F.J.*, 1911, 32).

* See Addenda, p. 577.

PROSECUTIONS FOR ORANGE WINE. *Wiltshire.* Salicylic acid 15 grains per pint. Fine £5 (*P.J.*, 1902, Aug. 16; *B.F.J.*, 1902, 202).

Haltwhistle. A glucose syrup containing about 0.5 % of citric acid, flavouring material, and 2.2 grains of salicylic acid per pint. It was labelled "British non-alcoholic orange wine." The Public Analyst stated that no orange juice was present, and that orange juice should be the basis of orange wine. For the defence it was maintained that in the B.P. definition of orange wine there was no mention of orange juice, and that the article practically agreed with that definition. Case dismissed (*P.J.*, 1915, Feb. 6; *B.F.J.*, 1915, 39).

London, Old Street. Proof spirit 20.6 %. The inspector asked for "non-alcoholic wine." The defendant pleaded that a mistake had been made, and was only ordered to pay costs (*Grocer*, 1926, Jan. 10).

PROSECUTIONS FOR GINGER WINE. *Longford.* Syrup flavoured with ginger and capsicum. Case dismissed as there was no standard (*P.J.*, 1903, Oct. 31).

Canterbury. Carbolic acid 0.028 %. Its presence was stated to be due to the combination of the chemicals used. Fine £1 (*Grocer*, 1921, Feb. 26; *B.F.J.*, 1921, 27).

Magherafelt. Salicylic acid 6.7 grains per pint. Fine 5s. and costs. On appeal to Quarter Sessions the magistrates' decision was reversed (*Grocer*, 1927, Nov. 5, 19).

Draperstown, Ireland. Salicylic acid 150 parts per million, contrary to the Preservative Regulations. Fine 10s. and costs (*Grocer*, 1930, April 26).

Moy, Ireland. Alcohol less than 1 %, when 12 % should have been present. Fine 2s. 6d. (*Grocer*, 1931, Feb. 28).

PROSECUTIONS FOR RAISIN WINE. *Eastbourne.* No trace of grapes or raisins; chiefly sugar, water and alcohol. Fine £3 (*Grocer*, 1907, Feb. 9; *B.F.J.*, 1907, 35).

Weston-super-Mare. Alcohol 12 % in "Non-alcoholic raisin wine." Fine £1 (*Grocer*, 1919, April 26).

London, Stratford. Salicylic acid $1\frac{1}{2}$ grains per pint, contrary to the Preservative Regulations. It was stated to have been taken into stock before the Regulations came in force. Paid costs (*Grocer*, 1929, March 30; *B.F.J.*, 1929, 49).

Lambeth. A solution of sugar in water coloured with an aniline dye. Labelled "British non-alcoholic raisin wine." Dismissed on proof of warranty. Subsequently the manufacturer was prosecuted for giving a false warranty. The defendant stated that the flavour was derived from essences, and evidence was given that it was of the usual commercial standard. The magistrate dismissed the case, having concluded that the article was what the consumer expected

to get (*Grocer*, 1929, March 16, April 13, 27; *Analyst*, 1929, **54**, 339; *B.F.J.*, 1929, 49, 60).

PROSECUTIONS FOR BLACK CURRANT WINE. *Norwich.* Salicylic acid 6 grains per pint. Fine 2s. 6d. (*Grocer*, 1912, Jan. 6; *B.F.J.*, 1912, 9).

Newcastle-on-Tyne. No black currant juice, being 21 % of sugar dissolved in water, with a small proportion of artificial flavouring and colouring materials, and $\frac{1}{2}$ grain of salicylic acid per pint. Dismissed on proof of warranty. Subsequently the manufacturers were summoned for giving a false warranty. The maker of the wine stated that to sugar, tartaric acid, fruit juices, and caramel, he added a black currant essence, which he assumed to be made from genuine fruit. The Bench dismissed the case, holding that when the manufacturers gave the warranty they believed it to be true (*Grocer*, 1925, Feb. 21; *B.F.J.*, 1925, 25, 64).

Mortlake. No definite evidence of the presence of black currant or other fruit juice, being an artificially coloured and flavoured solution acidified with a small proportion of organic acid. It was labelled "Fruit wine. Black currant flavour." Fine £10 (*Analyst*, 1927, **52**, 283).

PROSECUTION FOR RASPBERRY WINE. *Durham.* Not wine, but a syrup obtained from glucose, and coloured with an aniline dye. It was labelled "Fruit wine," though it contained no fruit. Defendant pleaded guilty and was fined £20. Subsequently he proceeded against his wholesale dealers at Glasgow for the fine and costs. The sheriff held that the plea of "guilty" barred the vendor from proceeding and dismissed the case. The sheriff considered that if he had contested the prosecution he should have been successful, both because there was no standard and because there was no prejudice to the purchaser (*P.J.*, 1904, June 25, Dec. 9; *B.F.J.*, 1904, 163).

PROSECUTION FOR ELDERBERRY WINE. *Durham.* No elderberry juice, but chiefly a solution of sugar, flavoured with cloves. The Government analysts reported that it did not correspond with genuine elderberry wine, but consisted of a coloured solution of sugar, flavoured with cloves, and containing 18 % of proof spirit. The Bench thought the wine was not what the purchaser would expect to get and had a right to have, and inflicted a nominal penalty of £1 (*P.J.*, 1904, July 2, Oct. 29; *B.F.J.*, 1904, 242).

PROSECUTION FOR GRAPE JUICE WINE. *Salford.* Grape juice less than 10 %. The defence stated that one-sixth of it was grape juice, and the remainder sweetened water coloured with burnt sugar. The stipendiary dismissed the summons as there was no standard by which he could judge if the proportion of grape juice was sufficient (*C. & D.*, 1880, Jan.).

CORDIALS

There is no sharp distinction between non-alcoholic wines and cordials, but as a rule the latter contain more solid extract. Many of the cordials in Hancock's Report (*loc. cit.*) contained more than 40 w/v of solid extract. Russell, in his 1928 and 1929 Bristol Reports, has given the composition of twenty-one unfermented wines and cordials (*Analyst*, 1929, **54**, 591 ; 1930, **55**, 685). Lime juice cordial is manufactured by mixing clarified juice with sugar and water, either cold or heated to 150° C.

In connection with the use of phosphoric acid in cordials, it is noteworthy that the seventeen samples of lime juice cordial, and the seven of lemon squash, given in Hancock's Report, were free from added phosphoric acid, the largest amount present being 0.02 w/v P_2O_5 ; also that a leading manufacturer advertises his lemon squash as containing "nothing but the pulp and juice of the fruit with only sugar added."

Hammond Smith had mentioned a substitute for citric or tartaric acid known as "phospho-citric acid," which consisted of 85 % of commercial phosphoric acid, and 15 % of citric acid. "Liquid tartaric acid" has been used as a synonym for commercial phosphoric acid (Arsenic Commission Report, II., 232).

During the years 1905-13, 32 % of the cordials examined in England and Wales were reported adulterated, and 33 % of the samples of lemon squash.

PROSECUTIONS FOR CORDIALS. *Southport.* "**Grape juice raspberry cordial.**" It contained no grape juice at all, but water 79.52 %, glucose 20 %, mineral matter 0.06 %, and colouring matter 0.42 %. Fine 10s. (*Grocer*, 1915, Oct. 30 ; *B.F.J.*, 1915, 239).

Kingston-on-Thames. **Raspberry cordial** containing 66 grains of phosphoric acid, and 5 grains of salicylic acid, per pint. Two samples of **black currant cordial** containing 67 grains of phosphoric acid, and 5 grains of salicylic acid, per pint. The Public Analyst stated that one fluid ounce of each of the samples contained nearly twice the maximum dose of phosphoric acid, and five times the dose of salicylic acid. The cordials were not prepared from fruit at all. The warranty on the bottle was not observed ; there was no such thing as "phospho-citric acid." Each vendor was fined £1. The wholesale dealers were summoned that they "did unlawfully abet, counsel or procure" the above defendants in the commission of the offences. They were fined £10 in each case (*B.F.J.*, 1921, 26).

Dover. **Strawberry cordial** containing phosphoric acid 35 grains per pint, sugar about 25 %, aniline dye, and possibly some extractive matter. For the defence it was stated that the cordial contained 1.2 % of strawberry essence, of which 2 oz. was equal to 3 lb. of

strawberries. A medical man considered there was no harm in the phosphoric acid ; it acted as a tonic and a thirst-quencher. Case dismissed (*B.F.J.*, 1921, 69).

London, Old Street. Cordials containing salicylic acid. **Raspberry** with 1·8 grains per pint, **orange** with 2·2 grains, **black currant** with 2·9 grains, **lemon** with 2·0 grains, and **vanilla flavour** with 2·5 grains per pint. Each vendor paid costs (*Grocer*, 1928, Jan. 14, ; *B.F.J.*, 1928, 14).

PROSECUTIONS FOR LIME JUICE CORDIAL. *Marylebone.* Phosphoric acid 40 grains per pint. The bottle was labelled "Schweppes' Lime Juice Cordial, prepared from the juice of the finest West Indian cultivated limes." The Public Analyst stated that the presence of phosphoric acid was unusual and unnecessary. Instead of the usual amount of 3 % of citric acid, there was only 1·3 %. It was not suggested that the addition was injurious. The defence argued that the inspector asked for "Schweppes' Lime Juice Cordial," and as he received that proprietary article, he was not prejudiced, whatever was its composition. The magistrate ruled against that point. The manufacturer gave evidence that the addition of phosphoric acid was necessary to take off the harsh flavour of lime juice. The magistrate dismissed the case, finding that the purchaser did not get an inferior article, and that there was no fraud ; he allowed 5 guineas costs to the defendant (*Grocer*, 1922, July 1 ; *B.F.J.*, 1922, 63).

Belfast. Salicylic acid 0·055 %, contrary to the Preservative Regulations. It was stated the article had been taken into stock before the Regulations came into force. Fine 5s. (*Grocer*, 1929, May 4).

PROSECUTIONS FOR LEMON SQUASH. *Westbury-on-Severn.* Salicylic acid 7 grains per pint. Fine £1 (*P.J.*, 1905, July 15).

Richmond-on-Thames. Phosphoric acid 1·20 %, salicylic acid 0·15 %. It was essentially a sweetened, flavoured, coloured and preserved solution of phosphoric acid. It was labelled "Absolutely pure, unadulterated lemon squash." The vendor proved a warranty, and his case was dismissed. Subsequently the wholesaler was prosecuted for giving a false warranty, and the manufacturer for giving a false trade description. The case against the wholesaler was dismissed, as he also proved a warranty, but the manufacturer was fined £20 (*P.J.*, 1920, Sept. 18 ; *Grocer*, 1920, Sept. 11 ; Nov. 13 ; *B.F.J.*, 1920, 85, 116).

Kingston-on-Thames. No trace of lemon juice, being an effervescent dilute solution of sugar, slightly acidified with phosphoric acid. It was labelled "Lemon squash, made from the fruit." The defendant said that lemon squash made from the fruit would not keep, but oil of lemon had been used. Fine 10 guineas (*Grocer*, 1921, April 2 ; *B.F.J.*, 1921, 37).

PROSECUTION FOR GINGER BRANDY. *London, Old Street.* Brandy absent. It was labelled "Ginger brandy (flavour), superior non-alcoholic." The case was dismissed and the defendant allowed 50s. costs (*Grocer*, 1929, March 9; *Analyst*, 1929, **54**, 288; *B.F.J.*, 1929, 46).

PROSECUTIONS FOR CHERRY BRANDY. *Chorley.* Salicylic acid 6 grains per pint. Labelled "Non-alcoholic cherry brandy." Fine £5 (*B.F.J.*, 1902, 138).

Brentford. Prosecution by the Board of Trade for false trade description. Evidence was given that it contained 0.25 % of proof spirit, and about $7\frac{1}{2}$ grains of salicylic acid per pint, and that original cherry brandy was brandy in which cherries had been steeped, which might contain 50 % of proof spirit. Another article, which ought to be called "cherry wine," was fermented cherry juice with brandy; it might contain 20 % proof spirit. Fine £5 (*B.F.J.*, 1921, 25).

MEDICATED WINES

Quinine wine, according to the B.P. of 1898 or 1914, is prepared by dissolving 20 grains of quinine hydrochloride in a pint of orange wine, and filtering, if necessary, after standing. No test is given for the finished wine. It has been suggested that filtration may remove part of the quinine as citrate or tannate. Orange peel contains little tannin, but if orange wine is stored in oak casks tannin is removed from the wood. It should be noted that the B.P. name is "Quinine Wine," not "*Orange* Quinine Wine." Both editions require that orange wine, and therefore quinine wine, be free from salicylic acid. The 1932 B.P. will contain no wines, and so the 1914 B.P. will remain the standard.

The following method may be used for its analysis. Take sp. gr. in 50 ml. bottle, evaporate to about one quarter volume, dilute to 50 ml. and take sp. gr. of extract. Dilute to about 100 ml., acidify with HCl, and extract with ether in a separator to remove oil. (One sample yielded 0.011 gm.) Add AmHO and extract with ether three times. The ethereal extract is allowed to evaporate and residue dried in water oven and weighed; for confirmation the alkaloid may be titrated. Other alkaloids may be removed by benzolated amylic alcohol. The ash may be determined on the aqueous residue.

Sp. gr. of alcohol = sp. gr. wine — (sp. gr. extract — 1.0).

7.2 % of the samples of "Quinine wine" examined in England and Wales during 1905–13 were condemned, and 14.9 % of the samples of "Orange quinine and quinine wine" during 1920–2.

Ipecacuanha wine is made from the liquid extract of ipecacuanha. According to the 1914 B.P., sherry was used, but in the 1932 B.P. 90 v/v alcohol, glycerine and water are to be used instead. A

direction is to be given that the resulting tincture should be dispensed when the wine is prescribed. It is probable, however, that in retail sales the wine of the 1914 B.P. will be preferred and usually sold. In these conditions, it is suggested that either the 1914 wine, or the 1932 tincture, may be sold when "ipecacuanha wine" is asked for.

Walton and O'Brien have given a colorimetric method for the determination of the alkaloids in the liquid extract of ipecacuanha (*S.P.A.*, 1931, **56**, 730).

During 1920-9, 12.6 % of the samples of ipecacuanha wine examined in England and Wales were condemned.

Medicated wines such as "**Beef and malt wine**," are probably taken chiefly for the alcohol present, and not for the small amount of food substances in them. Elsdon (*S.P.A.*, 1924, **49**, 210) has given analyses of a number of samples; they contained 16.1-20.4 v/v alcohol, and the largest proportion of nitrogen was 0.12 %. He also gave analyses of basis wines used in making them, and also of malt and meat extracts.

During 1923-30, 7.0 % of the "medicated wines" examined in England and Wales were reported adulterated.

PROSECUTIONS FOR QUININE WINE. *London.* The wine was labelled "One grain of sulphate of quinine in each wineglassful." The Public Analyst certified that $\frac{3}{5}$ grain was present in 2 oz. Evidence was given for the defence that sherry glasses contained $2\frac{3}{4}$ -3 $\frac{1}{2}$ oz. and port glasses 3 $\frac{3}{4}$ oz., and therefore the label was correct. Dismissed (*Analyst*, 1881, **6**, 173).

Trowbridge. No orange wine, but alcohol 4 $\frac{1}{2}$ % and colouring matter. Salicylic acid was present. Fine 30s. (*B.F.J.*, 1899, 93).

Highgate. Quinine deficient 25 %, 0.13 % being present instead of 0.18 %. The case was dismissed as the Public Analyst admitted that the wine might have been correctly prepared, and some of the quinine filtered out as tannate (*C. & D.*, 1900, Nov. 3, 10; *B.F.J.*, 1900, 376).

Dromore. Salicylic acid 5 grains per pint. After medical evidence for the defence, the case was dismissed (*P.J.*, 1916, Feb. 12).

Melton Mowbray. Quinine hydrochloride $\frac{1}{2}$ grain per oz., instead of 1 grain. An error in making the wine was admitted. Fine £5 and costs (*P.J.*, 1920, Aug. 21; *B.F.J.*, 1920, 80).

PROSECUTIONS FOR ORANGE QUININE WINE. *Kingston-on-Thames.* Strychnine hydrochloride 0.39 grain per oz. and no quinine. The defendant admitted that strychnine had been used instead of quinine. Fine £2 (*P.J.*, 1908, Aug. 1; *B.F.J.*, 1908, 143).

London, Old Street. Deficient of 95 % of quinine, being a solution of sugar containing a mere trace of quinine. Fine £5 (*Grocer*, 1929, March 9; *Analyst*, 1929, **54**, 288; *B.F.J.*, 1929, 46).

London, Old Street. Quinine 17 % deficient and entirely deficient in orange wine, no alcohol being present. Dismissed, as "*Orange*

quinine wine " was not mentioned in the B.P. (*P.J.*, 1929, April 27 ; *Analyst*, 1929, **54**, 339 ; *B.F.J.*, 1929, 69).

PROSECUTIONS FOR IPECACUANHA WINE. *Wolverhampton*. Salicylic acid 43 grains per gallon. Alcohol 14.5 % instead of 17 %. Defendant proved a warranty and was ordered to pay costs only. The wholesale dealers were subsequently prosecuted for giving a false warranty " full strength required by the B.P." and were fined £1 (*C. & D.*, 1896, June 27 ; *P.J.*, 1896, Oct. 17).

Lurgan. Alkaloids deficient 0.1 %. Subsequently the Government analysts reported that there was no deficiency, the alkaloidal extract being 0.115 gm. per 100 c.c. The case was dismissed and the defendant allowed £1 costs (*P.J.*, 1916, April 22).

PROSECUTION FOR BEEF AND MALT WINE. *Salford*. Not a beef and malt wine, but a flavoured cordial. It contained—alcohol 1.5 %, total sugars 21.8 %, salicylic acid 0.07 %, and other extractive matter 1.0 %, which included 0.01 % nitrogen. The amount of nitrogen indicated the possible presence of 0.2 % of a mixture of equal parts of meat and malt extracts. The vendor was fined £20 in each of three cases (*Grocer*, 1923, May 12 ; *Analyst*, 1923, **48**, 326 ; *B.F.J.*, 1923, 46).

PROSECUTION FOR LIEBIG'S BEEF AND MALT WINE. *Haltwhistle*. Proteids not more than one-fifth of what should be contained in the beef extract in a genuine sample, and practically no alcohol. The defence maintained that the inspector asked for a particular brand and obtained it ; he was therefore not prejudiced. Case dismissed (*Grocer*, 1924, April 5 ; *B.F.J.*, 1924, 43).

PROSECUTION FOR MEAT WINE. *Richmond*. Not more than 0.5 % of a meat extract containing 8 % of nitrogen. The case against the vendors was withdrawn, but the manufacturers were fined £10 for aiding and abetting the vendors, and £10 for giving a false label (*Analyst*, 1926, **51**, 460 ; *B.F.J.*, 1926, 90).

PROSECUTION FOR EXTRACT OF MEAT AND MALT WINE. *Salford*. Deficient of wine, no alcohol being present. It contained—total sugars 16.4 %, other extractive matter 2.4 %, including 0.06 % phosphoric anhydride, which indicated the possible presence of not more than 2 % of a mixture of meat and malt extracts. It should have contained at least 5 % of meat and malt extracts, and was deficient of 60 % of the minimum amount. The stipendiary dismissed the case as there was no legal standard of strength. On appeal, *Bowker v. Woodroffe* (1926), the High Court sent the case back to the stipendiary to look at the evidence to see what the standard was. The stipendiary restated his decision, and the case was again heard at the High Court, *Bowker v. Woodroffe* (1927). The Lord Chief Justice stated that it was the duty of magistrates to have regard to a minimum composition and to decide that upon any view of the minimum a compound falls short of it. The magistrate seemed

to think that, in the absence of a standard, anything could be sold as meat and malt wine, but it was his duty to say, on the evidence before him, that what was sold fell below the standard. The appeal was allowed, and on rehearing the vendor was fined £5. He subsequently proceeded against the manufacturer of the wine for damages and was awarded £205 10s. 6d. for breach of contract (*B.F.J.*, 1926, 86; *Analyst*, 1926, 51, 514; 1927, 52, 80, 341; *Grocer*, 1926, Aug. 28; 1927, June 25; 1928, May 12).

PROSECUTION FOR LIEBIG'S INVALID WINE. *Salford*. Not an invalid wine, but a flavoured artificial cordial, except for the presence of a trace of quinine. It contained 18·0 % of sugars (chiefly invert sugar), 1·5 % alcohol, 0·005 % of combined nitrogen, 0·004 % of combined phosphorus, 0·003 % of quinine sulphate, and 0·05 % of salicylic acid. It had not been made by the Liebig Company, but the use of that word suggested the presence of extract of meat, which, if present, did not exceed 1 part in 2,000. The vendor paid a nominal fine of 5s. and the wholesale dealers £20 for aiding and abetting (*Analyst*, 1925, 50, 403; *B.F.J.*, 1925, 66).

PROSECUTION FOR NON-ALCOHOLIC COCA WINE. *Leyland*. A flavoured syrup practically devoid of coca, containing 3·9 grains of salicylic acid per pint. Fine 2s. 6d. as it was considered to be a technical offence (*C. & D.*, 1904, Nov. 19).

CHAPTER XXVII

ACID FOODS. TARTRATES

Vinegar, definitions, brewed and artificial, standards, impurities, adulteration, decomposition. Table vinegar. Malt vinegar. Wine vinegar. Grape vinegar. Lemon juice. Lime juice. Lemonade powders. Tartaric acid. Cream of tartar and substitutes. Rochelle salt. Citric acid.

SOME time previous to 1889, a diluted and coloured acetic or pyroligneous acid was put on the market and sold as malt vinegar, or as vinegar. This substitution was made without any intimation to the public, and for some years public analysts were also unaware of it. In 1893, vendors of artificial vinegar were fined in Birmingham, and a conviction was confirmed by Quarter Sessions. As a result of this, and other prosecutions, there has been little substitution in Birmingham. Of 1,485 samples of "vinegar" examined 1894-1930, only twenty-three (or 1.5 %) were artificial vinegar.

DEFINITIONS. In 1908 Hamill made a Report to the L.G.B. on vinegar, its varieties, manufacture, and composition, and in the Annual Report for 1911 the L.G.B. suggested definitions "which might properly be adopted," and which largely followed Hamill's Report:—"Vinegar is a liquid derived wholly from alcoholic and acetous fermentation." "Malt vinegar is derived wholly from malted barley or wholly from cereals, the starch of which has been saccharified by the diastase of malt." "Artificial vinegar is any vinegar or substitute for vinegar containing or derived from any preparation containing any added acetic acid which is not wholly the product of alcoholic and subsequent acetous fermentation." All vinegars, natural or artificial, to contain at least 4.0 w/v of acetic acid. Unfortunately, the L.G.B. had not authority to make the suggested definitions legally binding. In 1926 a Vinegar Bill passed through Standing Committee, but did not become law. It defined "vinegar" in similar terms to those given above, and required "imitation vinegar" to be labelled as such, and kept in labelled receptacles. Malt vinegar was not mentioned.

BREWED AND ARTIFICIAL VINEGARS. The composition of the three different kinds of vinegar is shown in the following table, which is based on over 1,300 samples sold as "vinegar" and about 50 each of "malt" and artificial vinegar, bought in Birmingham 1893-1930:—

COMPOSITION OF BREWED AND ARTIFICIAL VINEGARS

ACETIC ACID, w/v	1.2-	3.8-	4.0-	4.2-	4.4-	4.6-	4.8-	5.0-	5.2-6.3	Total.
PERCENTAGE OF SAMPLES										
Malt vinegar	3	5	28	14	10	7	14	17	2	100
Vinegar	5	3	14	20	25	15	11	5	2	100
Artificial vinegar	21	—	10	14	8	19	6	14	18	100
TOTAL SOLIDS, w/v	0.2-	0.6-	1.0-	1.5-	2.0-	2.5-	3.0-	3.5-	4.0-5.2	Total.
PERCENTAGE OF SAMPLES										
Malt vinegar	0	0	5	19	22	34	7	6	7	100
Vinegar, 1893-	0	—	1	13	32	41	12	1	—	100
„ 1920-	0	—	8	32	21	32	6	1	0	100
„ 1925-30	0	—	8	42	19	4	14	12	1	100
Artificial vinegar	90	10	0	0	0	0	0	0	0	100
MINERAL MATTER, w/v	0.01-	0.1-	0.2-	0.3-	0.4-	0.5-	0.6	Total.		
PERCENTAGE OF SAMPLES										
Malt vinegar	0	7	26	34	20	7	6	100		
Vinegar	—	4	39	24	20	10	3	100		
Artificial vinegar	98	2	0	0	0	0	0	100		

The above figures for acetic acid show the reasonableness of the 4.0 w/v limit; the 8 % of the samples below this were either watered or decomposed samples of brewed vinegar. The figures show the uncertain composition of the artificial vinegars; 21 % were below 3.8 w/v of acetic acid, and 18 % had 5.2-6.3 w/v. These variations have been explained by the use of an acetic acid of unknown strength for dilution, to an error in calculation of the amount to be used, or to the use of an incorrect measure.

The total solids of the vinegars have been divided into three periods, which showed a progressive increase in the samples with low solids, owing to some alteration in materials and process of manufacture. The samples having less than 2 w/v were 14 %, 40 % and 50 %, respectively. All the artificial vinegars had less than 1.0 w/v, and nearly all of them had less than 0.1 w/v of ash.

The comparison of the brewed vinegars indicates that malt vinegars tend to be rather higher in acetic acid and solid matter, but otherwise there is not much difference in the figures.

The phosphates were determined in 134 vinegars, fifty malt vinegars, and in twenty-three artificial vinegars:—

PHOSPHATES (P_2O_5) IN VINEGARS

P_2O_5 , w/v	.002-	.01-	.02-	.03-	.04-	.05-	.06-	.07-	.08-	.09-.13	Total.
PERCENTAGE OF SAMPLES											
Malt vinegar	0	2	4	10	20	18	16	6	12	12	100
Vinegar	0	2	2	7	17	26	22	13	10	1	100
Artificial vinegar	87	13	0	0	0	0	0	0	0	0	100

The artificial vinegars were practically devoid of phosphates. The average percentage of nitrogen in malt vinegars was 0.080, in vinegars 0.069, and the artificial vinegars had a range 0.003-0.007.

The sp. gr. of vinegars depends both on the amounts of acetic acid and of total solids. Few brewed vinegars have sp. gr. less than 1.012 and artificial vinegars do not exceed 1.010. The figures depend on 594 samples of vinegar, 49 of malt, and 27 of artificial, vinegar.

SPECIFIC GRAVITY OF VINEGARS

(Sp. gr. \times 1,000)–1,000	5–	8–	12–	14–	16–	18–	20–	22–	26	Total.
PERCENTAGE OF SAMPLES										
Malt vinegar . . .	0	0	2	18	21	27	16	16		100
Vinegar	0	1	5	13	28	37	13	3		100
Artificial vinegar . .	30	70	0	0	0	0	0	0		100

Comparison of the above figures for malt vinegar and vinegar shows that the former tend to be somewhat higher in phosphates and nitrogen, but not markedly so. The explanation is probably that a large proportion of the samples sold in Birmingham as “vinegar” are really malt vinegar.

On the other hand, the only resemblance between the brewed vinegars and the artificial vinegar is in the acetic acid, the small proportions of other constituents being due to the colouring matter. A $\frac{1}{2}$ per cent. solution of caramel was found to yield 0.4 w/v of solids and 0.02 w/v of mineral matter.

Genuine vinegar is made by fermentation and acetification, and there is a considerable resemblance between the constituents present in vinegar and wine. Both contain ethers, aldehyde, organic acids, sugar, gum, nitrogenous and colouring matter, and mineral matter including phosphates. Vinegar contains acetic acid and a little alcohol, while wine contains alcohol and a little acetic acid. I have never heard it maintained that diluted and coloured alcohol can legitimately be sold as “wine,” and there is no more justification for selling diluted and coloured acetic acid as “vinegar.”

There is, of course, no objection to artificial vinegar of proper strength being sold as such; in some cases it has been claimed to be a better article than brewed vinegar, as not being liable to decomposition, but its makers show a strange reluctance to inform the public that they are buying the “better article”; and obtain increased profit by incorrectly selling it as “vinegar.” Hamill, however, considered it had harshness and pungency. In some cases manufacturers label the article for their own protection, and sell it to small shopkeepers who do not understand the position. One firm coined the term “Acetic vinegar.”

While some analysts do not agree with the above L.G.B. definitions, there is a strong body of opinion in support of it. In 1924, a few public analysts were invited to sign a statement that “Our experience shows that in many cases a coloured mixture of acetic acid and water is fraudulently sold in the place of genuine vinegar prepared by acetous and alcoholic fermentation.” It was

signed by sixteen analysts representing thirty-seven districts, which had a total population of over nine millions.

Owing to prosecutions, artificial vinegar makers have relinquished the use of the term "*malt vinegar*," but are prepared to defend its sale as "*vinegar*," which is of much greater importance, as comparatively few people ask for "*malt vinegar*." The advisability of prosecutions for its sale as "*vinegar*" has been previously discussed (p. 4).

STANDARDS. There have been many papers on the composition and analysis of vinegars. In addition to Hamill's Report, the following authors may be mentioned :—Hehner (*S.P.A.*, 1891, **16**, 81), Allen (*S.P.A.*, 1893, **18**, 241 ; 1894, **19**, 15), Ratcliff (*S.P.A.*, 1907, **32**, 85 ; 1909, **34**, 517), Fairley (*S.P.A.*, 1909, **34**, 515), Russell and Hodgson (*S.P.A.*, 1910, **35**, 346), Chapman (*S.P.A.*, 1912, **37**, 123), and Hodgson (*S.P.A.*, 1922, **47**, 254. Jamieson (*S.P.A.*, 1915, **40**, 106) has pointed out that vinegar made from malted *maize* gives low analytical figures. Genuine distilled vinegars contain furfural, which is absent from acetic acid itself (see Lampitt, Hughes and Trace, *S.P.A.*, 1927, **52**, 260).

The author, from his own experience and consideration of published analyses, and of a number for which he is indebted to W. T. Rigby, suggests the following limits in addition to 4.0 w/v of acetic acid :—

Vinegar should have at least 1.5 w/v of total solids, and 0.18 w/v of mineral matter. Malt vinegar should, in addition, have at least 0.05 w/v of P_2O_5 and 0.04 w/v of nitrogen. In some prosecutions the very low standards of 0.03 w/v for P_2O_5 and nitrogen have been taken.

Low phosphates and nitrogen may not be due to added acetic acid, but to dilution with water. In such a case the calculation of the amount of malt vinegar may be facilitated by expressing the phosphates and nitrogen in relation to 100 parts of original solids, and not on 100 parts of vinegar, as suggested by Hehner (*opus cit.*). It is assumed that the acetic acid is derived from glucose.

$$\text{Original solids (w/v)} = \text{Total solids (w/v)} + \frac{180 \times \text{acetic acid (w/v)}}{120}$$

On this basis, the original solids of malt vinegar should contain at least 0.5 % each of P_2O_5 and nitrogen. In the analyses from which these figures are deduced, the alteration of the basis of calculation has more effect on one constituent than on the other.

Another comparison which may be used is the percentage of P_2O_5 in the ash of the vinegar ; it should be at least 14 %.

IMPURITIES. Of twenty-three samples tested for *arsenic*, three contained only 0.1–0.2 parts per million. Twenty-two samples tested for *lead* had an average of 1.2 parts per million. The samples were systematically tested for *boric acid*, but only one sample each

of malt vinegar, vinegar, and artificial vinegar contained it, the quantities varying from $1\frac{1}{2}$ to 3 grains per gallon. *Salicylic acid* was also regularly tested for, but not detected. *Sulphur dioxide* was not detected in forty samples tested, and two others had doubtful traces. Small quantities may be normal (Cox, *S.P.A.*, 1927, 52, 397). One sample which had 0.63 % of mineral matter was found to contain chlorine equivalent to 0.37 % of salt; fourteen others averaged 0.07 % of salt. One sample which gave a reddish ash had 0.02 % of iron, eight others averaged 0.006 %.

In only one sample was free *sulphuric acid* detected, and that was an artificial vinegar. In 1818 it was believed that sulphuric acid was necessary to preserve vinegar, and 0.1 % by weight was allowed to be added by the Vinegar Act of that year. The Act was repealed in 1861. (*Cf.* Edmunds, *B.F.J.*, 1900, 212).

A possible impurity is fluoride, which has been offered as a cask cleaner. C. A. Mitchell has called attention to the addition of ammonium salts to spurious vinegars, presumably to make them more likely to be passed as brewed vinegar (*S.P.A.*, 1931, 56, 178).

ADULTERATION. In 1893, no less than 15.5 % of the samples examined in England and Wales were adulterated. During 1894–1907, the proportion decreased to 6.3 %, during 1908–13 it increased to 11.0 %, and again decreased to 7.3 during 1919–30.

DECOMPOSITION. Deficiency in acetic acid strength when a vendor is prosecuted is sometimes attributed to evaporation or decomposition of the vinegar.

The effect of evaporation was tested by putting vinegar into a porcelain dish and allowing it to evaporate; it was weighed and titrated after one and four days. Dilute acetic acid was treated similarly.

EFFECT OF EVAPORATION

Exposure, days	Vinegar.			Dilute Acetic Acid.		
	0	1	4	0	1	4
Loss of weight, %	—	10	46	—	10	47
Acetic acid, w/v.	3.84	4.02	4.68	4.14	4.23	4.68

In each case the liquid lost weight and became stronger, owing to water evaporating more rapidly than acetic acid.

Good vinegar may be kept in corked bottles partly filled for a long time with little change. In the examples below, (I.) represents the original figures (w/v) for the three vinegars, and (II.) the corresponding results twenty-seven months later, after decantation from small sediments. There had probably been some evaporation.

VINEGARS KEPT FOR TWENTY-SEVEN MONTHS

Constituent.	Acetic Acid.		Total Solids.		Ash.		P ₂ O ₅ .		Nitrogen.	
	I.	II.	I.	II.	I.	II.	I.	II.	I.	II.
Analysis										
Vinegar, A.	4.08	4.02	3.08	2.58	.33	.33	.080	.077	.066	.069
„ B.	4.74	4.59	4.30	3.67	.34	.36	.105	.095	.084	.091
„ C.	4.86	4.62	4.01	3.42	.41	.40	.131	.121	.086	.095

The following are examples of samples measuring about 3 oz. which were examined on receipt, and again after keeping about a month in the corked bottles :—

ACETIC ACID (w/v) IN VINEGARS KEPT FOR A MONTH

Sample	D	E	F	G	H	J	K
Original strength . . .	3.30	2.14	3.12	1.50	1.19	4.5	4.1
About a month later . .	3.33	2.21	2.91	1.35	1.11	3.9	2.3

Samples D and E had gained slightly in strength, F-H had lost slightly. J had lost 0.6 w/v, there being a growth of mother of vinegar (*Bacterium xylinum* : A. J. Brown, *J.C.S.*, 1886, **49**, 432) in the bottle. K had lost 1.8 w/v ; in this case there was a white filmy growth on the sides of the bottle.

In the above cases the bottles were kept well corked ; two others lost nearly all their strength by keeping in uncorked bottles. One sample fell from 3.03–0.66 w/v, and the other 2.79–0.50 w/v.

The effect of various organisms taken from decomposed samples on a sterilised vinegar containing about 4 % of acetic acid was tried. In fifteen weeks, mother of vinegar, yeast, *Penicillium glaucum*, and *Aspergillus niger* had no effect, but a white film organism taken from a beer reduced the strength to 0.3 %. The samples were then diluted with water to about half strength, and in seven weeks more the first three did not lose more than 0.1 % of acetic acid. The *Aspergillus niger*, however, reduced the strength to 0.6 %.

An informal sample of vinegar contained only 3.5 % of acetic acid, and a subsequent formal sample taken a week later had 2.9 %. As the quantities of total solids and mineral matter in the two samples were identical, it was not probable that the difference in strength was due to added water. The barrel from which it had been taken admitted air owing to the shrinking of its staves. F. D. Ratcliff suggested to me the vinegar at the surface was weaker owing to the growth of a white micro-organism there in the presence of too much air, and that the greater strength of the first sample was due to it being taken further from the surface than the second sample.

To investigate the question, vinegar containing 4 % of acetic acid was put in a cylindrical separator about 7 inches deep, and heavily infected with the organism in question. After a month the surface vinegar contained only 1.5 % and that at the bottom 3.7 % of acetic acid. Vinegar contained in a nearly empty cask, particularly if there is ventilation, is therefore liable to decomposition.

Some experiments on the alteration of strength of vinegar stored in wooden casks have been recorded by Behre (*Analyst*, 1912, **37**, 23). F. D. Ratcliff found that a cask of vinegar lost weight on keeping, while the vinegar in it became stronger.

Occasionally deficiency in strength is attributed to the presence of the vinegar eel. This did not appear probable as six of the twelve samples in which they were observed contained 4.4–5.5 % of acetic acid. Vinegars containing 4.6 and 3.9 % of acetic acid, respectively, were infected with them. They were alive a month later, but the strength of the vinegar was unchanged. They are about 500μ long and about 20μ broad. Peters has investigated the bionomics of them (*Analyst*, 1928, **53**, 661); he states that they are non-pathogenic to man, and that they slightly increase the acidity of vinegar. At a prosecution for their presence, evidence was taken that they were not injurious, and the case was dismissed (*Analyst*, 1880, **5**, 71, 83).

On one occasion trouble was caused by frost. A cask of vinegar became partly frozen. Vinegar, concentrated by the separation of ice, was taken from the bottom. Later on there was a complaint as to the weakness of the remaining liquid, which had been diluted by the melted ice.

Table Vinegar. In 1926 for the first time a sample of "table vinegar" was submitted to me for analysis, and enquiries were made as to the meaning of the term. The replies of nine vinegar brewers stated that the term was instituted many years ago to describe a superior kind of malt vinegar, that it has been much less used since the war, that it should be a brewed vinegar, and that it is used "to sell to the public an artificial vinegar under the impression that they are buying a malt vinegar." The opinion of retailers was that "table vinegar" meant a superior kind of malt vinegar for table use, and not an artificial vinegar. The Birmingham Grocers' Association authorised its President and Vice-President to give evidence to that effect in a prosecution for the sale of artificial vinegar as "table vinegar," but the magistrates convicted without their evidence being called.

White Wine Vinegar. The term is a misnomer, vinegar which is white is not wine, and wine vinegar is not white. There has been a difficulty in getting genuine wine vinegar, and dilute acetic acid has been widely sold in its place. About 1895 there were prosecutions for such substitution, and in 1896 there was correspondence on the subject in the *Pharmaceutical Journal* (Jan. 18, 25; Feb. 8). A. H. Allen read a paper at the British Pharmaceutical Conference in that year (p. 321, abstract, *Analyst*, 1896, **21**, 253), and a discussion followed. See also Proctor (*P.J.*, 1896, Aug. 16). Allen suggested that the substitute should be labelled "Distilled Vinegar, commonly called White-wine Vinegar." Genuine wine vinegar should contain 6 % of acetic acid and also tartrate. Farnsteiner has studied the changes which take place during the acetous fermentation of wine (*Analyst*, 1899, **24**, 151).

METHODS OF ANALYSIS. *Acetic acid* is conveniently determined by adding 15 ml. N/2.NaOH to 10 ml. of vinegar in a

white dish, adding phenol-phthalein as indicator, and titrating back with $N/2.HCl$. The *total solids* may be determined on 5 ml. by drying three hours in a flat-bottom porcelain or platinum dish. They may retain a trace of acetic acid (Russell and Hodgson, *S.P.A.*, 1910, **35**, 348). The *mineral matter* should be determined on 50 ml. if available.

Free Mineral Acids. The ash of a normal vinegar is alkaline, the amount of alkalinity depending on the water and other ingredients used in brewing it. Small additions of acid may be insufficient to neutralise such alkaline substances, and the ash may still be alkaline. An alkaline ash, however, indicates that the amount of added acid, if any, is small. The presence of free sulphuric acid will make the total solids black by charring them. Its presence may be confirmed by suspending a strip of filter paper (say $\frac{1}{2} \times 12$ inches) so that one end is in the vinegar, leaving all night, and drying the strip in the water oven for a short time. The liquid rises by capillarity, and the sulphuric acid remains at the upper part and chars the paper on drying. To calculate free sulphuric acid from a determination of total sulphate is obviously incorrect. Methods for determination of mineral acids have been given by *Hehner* (*S.P.A.*, 1877, **1**, 105), *Schidrowitz* (*S.P.A.*, 1903, **28**, 233; 1907, **32**, 3), and *Ratcliff* (*S.P.A.*, 1907, **32**, 82).

Reif (*Analyst*, 1926, **51**, 41) has given a test for tannin as characteristic of fermentation vinegars, and *Brode and Lange* (*Analyst*, 1909, **34**, 157) have described methods for determining various constituents of vinegars.

PROSECUTIONS FOR VINEGAR. *Birmingham.* Pyroligneous acid 70 %. Fine £1. The defendant appealed to Quarter Sessions, and much expert evidence was given. The Recorder thought that references to acetic acid in Excise Acts did not prove that acetic acid was recognised by law as vinegar. Secondly, although the mixture had been sold for a number of years as "vinegar," the public and analysts had not, until recently, been aware of the change. Thirdly, in point of fact, the substance was not vinegar. The British Pharmacopœia defined vinegar as prepared by fermentation, and evidence had been given that acetic acid was "as different from vinegar as silent spirit is different from wine." The appeal was dismissed with costs (*F. & S.*, 1893, May 27, July 8; *Analyst*, 1893, **18**, 208).

Birmingham. Pyroligneous acid 80 %, sulphuric acid 0.14 %. Fine 5s. (*F. & S.*, 1893, May 20).

Sheffield. Dilute acetic acid 80 %. The Government analysts reported "that the acetic acid derived from wood did not exceed a fourth of that present in the vinegar. The maker then admitted adding acetic acid. Fine £5 (*C. & D.*, 1894, June 9, July 7).

Swansea. Iron in solution 15 grains (per gallon?). The Bench

found that the iron present was accidental and dismissed the case (*F. & S.*, 1896, Nov. 28).

London, Marlborough Street. Water 50 %. Fine £3 (*B.F.J.*, 1899, 221).

London, Marlborough Street. Sulphuric acid 0.15 %. An analyst for the defence found phosphoric acid, but no sulphuric acid. The Government analysts were of opinion that 0.14 % of sulphuric acid had been added to the vinegar. Fine £5. There were three other similar cases (*B.F.J.*, 1900, 29, 35, 42, 212).

Howden. Vegetable *débris* and decomposing organic matter 20 parts per 100,000. Fine £1 (*Grocer*, 1911, May 6; *B.F.J.*, 1911, 100).

St. Helens. Copper 0.75 grain per pint. The Government analysts confirmed this. The Bench considered the impurity was due to an accident and ordered payment of costs (*Grocer*, 1913, March 1; *B.F.J.*, 1913, 80).

Birmingham. Impure acetic acid 3.4 %, solid colouring matter 0.4 %. The Bench accepted the suggested standard of at least 4 % of acetic acid and ordered payment of costs (*P.J.*, 1917, Jan. 27).

Southampton. Acetic acid 2.91 %, being deficient of 27.2 %, as normal vinegar contains at least 4 %. Case dismissed as there was no legal standard for vinegar. On appeal, *Newman v. Robinson* (1917), the High Court directed the magistrates to convict (*B.F.J.*, 1917, 100, 154).

Birmingham. Acetic acid 2.2 %, being 45 % deficient in strength. Fine £5 (1918 Report).

Lindsey. "Deficient in acetic acid to the extent of 16 %. According to the recommendations of the Local Government Board," vinegar should contain not less than 4 % of acetic acid. Dismissed. On appeal, *Cook v. Jonnson* (1918), the High Court decided that the Public Analyst had a perfect right to express his opinion in the way he had done, as was decided in *Harrison v. Richards* (1881), (*B.F.J.*, 1918, 50).

Newcastle-on-Tyne. Tartaric acid 3.8 %, water, colouring matter, etc., 96.2 %. The defence was that it was sold as a substitute. Paid costs (*Grocer*, 1919, Jan. 18; *B.F.J.*, 1919, 27).

London, Old Street. Added water 4 %. The manufacturer stated he had made the vinegar himself from the requisite amount of (untested) acetic acid. Fine and costs £12 (*Grocer*, 1920, Jan. 10).

Birmingham. Acetic acid 2.9 %. It was part of the last half gallon in a 12½-gallon cask. The defendant undertook to order 6-gallon casks in future and was only ordered to pay costs (*B.F.J.*, 1923, 110, *Analyst*, 1923, 48, 544).

Londonderry. Lead 5 parts per million. The inspector cut the lead capsule covering the neck of the bottle, and poured the sample into another bottle. The defence submitted that the contamination

was due to the method of sampling. The case was withdrawn as the manufacturers had ceased to use such capsules (*P.J.*, 1926, June 19 ; *B.F.J.*, 1926, 80).

Manchester. Artificial vinegar. Four defendants were each ordered to pay costs (*Grocer*, 1927, Jan. 29).

Birmingham. Artificial vinegar containing 3.3 % of acetic acid. The first sample was taken from a small shop, and, at the request of the owner, a second was taken in course of delivery from the makers. The summons against the shopkeeper was withdrawn, and each of the three brothers who sold the second sample was fined £1 (1927 Report).

London, Tower Bridge. Zinc 0.51 %. The vendor was unable to explain the impurity. The vinegar had not been kept in a zinc tank, nor in a cheap enamel vessel. Paid costs (*Grocer*, 1928, March 17).

Plymouth. Acetic acid deficient 23 %. The defendant admitted a mistake in mixing it. Fine and costs £1 (*B.F.J.*, 1929, 50).

Hull. Acetic acid only 3.0 %. The vinegar manufacturer, who sold the barrel, said the vinegar had frozen, and suggested the acid had gone into the wood. Fine £1 (*Grocer*, 1929, April 27).

Greenwich. Water in excess 12 %. It was suggested that melting snow had percolated into the barrel, which was kept in the garden. Paid costs (*Grocer*, 1929, June 15).

London, Old Street. Excessive water 35 %. It had been prepared from "essence." Paid costs (*Grocer*, 1929, Sept. 14).

Blackpool. Diluted acetic acid. The bottle was labelled "Finest quality of table vinegar for pickling and other purposes." Evidence was given that the article was superior to brewed vinegar as it did not decompose. Case dismissed (*Grocer*, 1930, Aug. 30 ; *P.J.*, 1930, Sept. 6 ; *B.F.J.*, 1930, 100).

Godstone. Acetic acid 3.4 %. The defendant said it was wood vinegar he had made from "essence." Fine and costs £1 (*Grocer*, 1931, July 25).

Liverpool. Giving a false warranty with vinegar that contained only 2.4 % of acetic acid. Fine £2 (*Grocer*, 1931, Aug. 8).

London, Old Street. Acetic acid deficient 12.5 %. Evaporation was suggested as an explanation of the deficiency. Fine £5 (*Grocer*, 1931, Nov. 7).

PROSECUTIONS FOR MALT VINEGAR. *Bristol.* Diluted acetic acid and colouring matter. The barrel from which it was taken was labelled "Pure Malt Vinegar." Fine 10s. (*F. & S.*, 1893, April 29).

Washington. Added water 50 %. Fine 15s. (*F. & S.*, 1894, 75).

London, Marlborough Street. Sulphuric acid 0.121 %, while it should not exceed 0.022 %. The manufacturer stated that no free sulphuric acid had been added, but that the "sulphuric acid" was due to the calcium sulphate present in the brewing water, which was

similar to that used for brewing Burton ales. Case dismissed (*B.F.J.*, 1900, 114, 145, 213).

North London. "Vinegar other than malt vinegar." The magistrate remarked that the certificate did not say whether the parts shown on it (acetic acid, non-volatile organic matter, mineral matter) were foreign to malt vinegar. The case was adjourned so that the Public Analyst could give evidence on the point. Fine £2 (*Grocer*, 1909, Sept. 4; *B.F.J.*, 1909, 168, 189).

Nuneaton. Vinegar not derived from malted barley or cereals 30 %, acetic acid 4.0 %, nitrogen 0.022 %, phosphoric acid (P_2O_5) 0.024 %. The Government analysts found nitrogen 0.019 %, and phosphoric acid (P_2O_5) 0.020 %, and were of opinion that the vinegar not derived from malt was not less than one-third of the sample. Fine £7. The defendant appealed, *Grimble v. Preston* (1914), and it was argued, among other things, that there was no evidence upon which the justices ought to have convicted, as there was no legal standard as to the proportion of phosphoric acid that vinegar ought to contain. The appeal was dismissed (*B.F.J.*, 1913, 93, 111, 211).

Leeds Assizes. One vinegar brewery company sued another for damages for libel, in that leaflets had been distributed announcing the fact that a customer of the former company had been fined for selling artificial vinegar as malt vinegar. The damages awarded were one farthing (*B.F.J.*, 1915, 58).

Warrenpoint. Lead 17 parts per million. The manufacturers suggested that particles of the tinfoil capsule had fallen into the bottle. Case dismissed (*Grocer*, 1925, June 6; *B.F.J.*, 1925, 70).

London, Old Street. Sulphur dioxide 50 parts per million. The brewer, who admitted using a cleansing solution, was ordered to pay 5 guineas costs (*Grocer*, 1928, March 3).

Birmingham. Artificial vinegar containing only 3.6 % of acetic acid. Fine 10s. (*Grocer*, 1929, Jan. 12).

London, Lambeth. Deficiency in acetic acid 63 %. The cask from which it was taken was almost exhausted (*Grocer*, 1929, March 16).

London, Old Street. Sulphur dioxide 150 parts per million. A powder, sold by a traveller to clarify the vinegar, had been added. Dismissed (*Grocer*, 1929, July 20, 27).

Keighley. Artificial vinegar, at least 75 %. The cask was labelled "Malt vinegar, Guaranteed absolutely pure, Specially blended." Fine £1 (*B.F.J.*, 1931, 59).

Grimsby. Artificial vinegar. Fine 5s. (*Grocer*, 1931, May 30).

Fenton. Artificial vinegar made from acetic acid. The cask was marked "Absolutely pure vinegar, and may be sold as such under the Food and Drugs Act. It is sold as pure vinegar without any other description." Fine £2 (*Grocer*, 1931, Nov. 14).

PROSECUTIONS FOR TABLE VINEGAR. *Birmingham.* Artificial vinegar. The magistrates decided that it was "not the article asked for," and fined the vendor 5s. (*Analyst*, 1927, **52**, 29).

Atherstone. Artificial vinegar 100 %. Evidence was given that 9s. had been paid for a 6-gallon barrel, that pure malt vinegar could be bought for 8s. 6d., and that artificial vinegar was usually quoted at 4s. for 6 gallons. Case dismissed. On appeal, *Preston v. Jackson* (1928), the High Court ruled that, upon the uncontradicted evidence of the Public Analyst, the only course open to the justices was to convict the respondent. The vendor was subsequently fined £2 (*Grocer*, 1928, Sept. 15, Dec. 22; *B.F.J.*, 1928, 99, 114; 1929, 20; *Analyst*, 1928, **53**, 592; 1929, **54**, 32).

Cork. Acetic acid 1.5 % and 2.25 %, respectively, instead of 4 %. The vendor was fined 5s. in each case (*Grocer*, 1930, Dec. 27).

Wolverhampton. Artificial vinegar. It was labelled "Table vinegar, pure and wholesome." The retailer, who did not know it was not vinegar, was fined 30s. (*Grocer*, 1930, July 12).

Solihull. Artificial vinegar 100 %. Evidence was given that about 6d. per pint was charged for it, and that genuine malt vinegar was bought from the same vendor at 3d. per pint. Fine 10s. (*North Birmingham News*, 1932, Feb. 20).

PROSECUTIONS FOR WHITE WINE VINEGAR. *Enniskillen.* Sulphuric acid 0.515 %. Fine £5 (*F. & S.*, 1893, Dec. 9).

Burslem. Diluted acetic acid. The defence was that the article was of the usual composition. Fine £2 (*C. & D.*, 1895, May 25).

Southampton. Diluted distilled acetic acid. The Government analysts reported that the article was "distilled vinegar, commercially known as white wine vinegar, and that it does not consist of diluted acetic acid." Case withdrawn (*F. & S.*, 1895, July 6, Aug. 3).

Hanley. Diluted acetic acid. Evidence was given that the manager who sold the article had acted contrary to the special printed instructions of the company. Convictions followed, and on appeal to Quarter Sessions the fines were reduced, the company paying £5 and the manager £2 (*F. & S.*, 1896, Jan. 4, April 18).

PROSECUTIONS FOR GRAPE VINEGAR. *Liskeard.* Wood vinegar 100 %. Evidence was given that it contained no phosphate, tartrate or inositol, which would be found in a grape product. The manufacturer was fined £1, and also £1 for a false label. A retailer was fined £1 for the sale of an article containing only 3.2 % of acetic acid, instead of 4 % (*B.F.J.*, 1931, 96; *Analyst*, 1931, **56**, 660; *P.J.*, 1931, Sept. 19).

PROSECUTION FOR FRENCH WINE VINEGAR. *Swainsthorpe.* Distilled spirit vinegar of low strength. Fine £1 (*Grocer*, 1906, Jan. 13).

PROSECUTION FOR WOOD VINEGAR. *London, Marylebone.*
Acetic acid 3·5 %, instead of 4 %. Fine £2 (*Grocer*, 1929, March 30).

LEMON JUICE

Lemon juice is required by the 1914 B.P. to contain 7–9 w/v citric acid, and to have sp. gr. 1·030–1·040. The dry residue should not yield more than 3 % of ash. The acidity of Hancock's four samples of lemon juice varied 6·5–8·1 w/v, expressed as citric acid sp. gr. 1·030–1·038, extract 8·1–10·3 w/v, ash 0·33–0·55 w/v, and phosphoric anhydride (P_2O_5) 0·01–0·02 w/v. In each case the ash was more than 3 % of the dry extract. All four contained sulphur dioxide, two boric acid also, and one salicylic acid. Detailed analyses of lemon juice have been given by Borntraeger (*Analyst*, 1898, **23**, 176), Lührig (*Analyst*, 1906, **31**, 233), Huerre (*Y.B.P.*, 1919, 147), and Azadian (*Analyst*, 1925, **50**, 626).

G. D. Elsdon, in his 1925 Salford Report, has made some apt remarks on the way in which a food may be gradually deteriorated :—

“Lemon juice is a natural article—the first stage of its adulteration is in using a solution of citric acid which has been prepared from some fruit, and flavouring it with distilled oil of lemon. Seeing that according to this, citric acid may be used, it is only a short step to substitute tartaric acid, which, of course, does not exist in lemons, and after this, the next stage, which is still more objectionable, is the use of phosphoric acid. The final result of what has been described as a series of ‘justifiable modifications of formulæ’ is a substance which bears no real relationship whatever to the substance it is intended to replace.”

For the detection of *artificial* lemon juice, Spaeth (*Analyst*, 1901, **26**, 269) has pointed out the usefulness of the relation between the ash and its alkalinity. Natural lemon juice contains about 0·4 % of ash, and it requires about 6 ml. of N.NaOH to neutralise 100 ml. Citric acid solutions have neither ash nor alkalinity. Tillmans and Kiesgen (*Analyst*, 1927, **52**, 417) utilise, for this purpose, the increase of alkalinity to phenol phthalein produced by formalin.

LIME JUICE

The total acidity of twelve samples, reported on by Hancock (Ministry of Health Report, No. 24), varied 6·1–9·2 w/v, expressed as citric acid. The extract varied 7·97–11·34 w/v, the ash 0·26–0·44 w/v, the phosphoric anhydride (P_2O_5) 0·01–0·03 w/v, and the sp. gr. 1·028–1·043.

Experiments made at the Dominica Botanical Station proved that a good average juice, containing 7·7 w/v of citric acid, when kept in a cask for six weeks, only lost 1·7 % of its strength. Weak juice deteriorated more rapidly (*P.J.*, 1912, July 13).

Of the samples examined in England and Wales, 1905-13, 29 % were reported adulterated.

PROSECUTIONS. *Bournemouth.* Sugar 30.9 %, and only 40 % of lime juice, the citric acid being only 1.9 %. It was a lime juice cordial, though labelled "Pure lime juice." Paid costs (*B.F.J.*, 1912, 175; *Grocer*, 1912, Aug. 17).

Bournemouth. Excess water 95 %, only 0.15 % citric acid being present. It was slightly acidulated with phosphoric acid, and coloured with coal-tar dye. Fine £5 (*Grocer*, 1918, Oct. 26; *B.F.J.*, 1918, 114).

Kingston-on-Thames. Phosphoric acid 1.16 %, salicylic acid 0.14 %. It was not lime juice, but a flavoured, coloured, sweetened solution of phosphoric acid. The vendor was fined 5s., and the wholesaler £20 and 10 guineas costs for aiding and abetting and £1 for applying a false trade description (*B.F.J.*, 1920, 117; *Grocer*, 1920, Nov. 27).

LEMONADE CRYSTALS. LEMONADE POWDERS

"Lemonade powders" have been described by Hammond Smith (Arsenic Commission Report, ii., 242) as being composed of about equal parts of citric or tartaric acid, and sugar.

Two Birmingham samples of "lemonade crystals" were examined in 1913. One was composed of about 30 % of tartaric acid, and 70 % of sugar. The other contained about 30 % of a mixture of citric and tartaric acids, with sugar and about 7 % of glucose. The rapid inversion of the sugar by the acid made polarisation difficult. The samples contained a small amount of ash, 0.17 % and 0.12 %, respectively, and each had about 9 parts of lead per million.

Of the samples of lemonade powders or crystals examined in England and Wales, 1920-30, 7.2 % were adulterated; the adulterated samples were mostly bought in 1926 and 1928.

PROSECUTIONS FOR LEMON POWDER. *Kingston-on-Thames.* Tartaric acid 49.2 %, cane sugar 49.7 %, moisture, traces of artificial colouring matter, and flavouring essence, which might have been essence derived from the lemon, 1.1 %. Fine £20 for false trade description, "Messina lemonade, made entirely from fresh lemons" (*B.F.J.*, 1921, 86).

Richmond-on-Thames. Tartaric acid 7.9 %, cane sugar 90.7 %, moisture 1.4 %. There were traces of colouring matter not from lemons, and traces of flavouring essence, which might have been derived from lemons. The packets were labelled "Glass lemon produced by a new scientific process of concentrating the actual lemon," and that it would produce "a perfect beverage made with the actual fruit." The vendors were fined £10, for selling the article

to the prejudice of the purchaser, and the manufacturers £20, for the use of a false trade description, and the same amount for aiding and abetting (*Grocer*, 1921, Nov. 12; *B.F.J.*, 1921, 108).

PROSECUTION FOR LEMONADE CRYSTALS. *Kingston-on-Thames.* Tartaric acid 30·8 %, cane sugar 68·6 %, moisture and flavouring essence, with traces of artificial colouring matter 0·6 %. No citric acid was detected. The packets were labelled "Lemonade crystals, containing all the virtues of fresh lemons." The Medical Officer of Health said the article did not contain the important anti-scorbutic properties of fresh lemons, and that tartaric acid was an irritant, and inferior to citric acid. For the defence it was stated that the flavour was really got from lemon peel, so that the crystals did contain one of the virtues of the lemon. The Bench dismissed the case, being of opinion that no prejudice to the purchaser had been proved (*P.J.* and *Grocer*, 1922, Feb. 18; *B.F.J.*, 1922, 15).

TARTARIC ACID

The B.P. gives a limit of 20 parts of lead per million. Only two of the sixty-one Birmingham samples examined 1916–29 exceeded this limit.

LEAD IN TARTARIC ACID (sixty-one samples)

Parts of lead per million	.	.	0–	6–	11–	16–	21, 27	Total.
Percentage of samples	.	.	35	38	13	11	3	100

None of the samples exceeded 1 part of arsenic per million. The limit for ash of the 1914 B.P. (0·1 %) was frequently exceeded.

ASH IN TARTARIC ACID (sixty-one samples)

Ash, %	.	.	0–	0·11–	0·21–	0·31–0·37	Total.
Percentage of samples	.	.	38	29	27	6	100

During 1905–13, of the samples examined in England and Wales, 6·4 % were reported adulterated, and during 1920–30, the proportion was 1·1 %.

PROSECUTIONS. *Devizes.* Cream of tartar 51·8 %, calcium tartrate 3·2 %. Fine £1 (*F. & S.*, 1896, March 28).

Glasgow. Tartaric acid 93·75 %, instead of 99·75 % according to the B.P. The vendor was a grocer, who said he sold it for household purposes. Fine 2s. 6d. (*P.J.*, 1902, Dec. 20).

Swindon. Arsenic at least 7 parts per million. The Government analysts found 14 parts. Fine 10s. (*P.J.*, 1904, June 25; *B.F.J.*, 1904, 160).

Bilston. Arsenic $\frac{1}{2}$ grain per lb., lead $\frac{1}{10}$ grain per lb., to the prejudice of the purchaser. Fine £2 and costs (*B.F.J.*, 1907, 216).

Southport. Cream of tartar 23.36 %. The defendant admitted carelessness in filling up a drawer. Fine £3 and costs (*P.J.*, 1921, Dec. 31).

CREAM OF TARTAR. PURIFIED CREAM OF TARTAR

The composition and analysis of cream of tartar has been discussed in considerable detail by Allen (*S.P.A.*, 1880, **5**, 114; 1896, **21**, 174). A Report by Macfadden to the L.G.B. in 1907 gave an account of its manufacture, and recommended limits of 20 parts of lead, and 1.4 of arsenic, per million.

There has been a great improvement in the quality of cream of tartar. The 1885 B.P. required a limit of about 92 % purity, but about 60 % of the Birmingham samples examined 1887-97 failed to reach this low standard. The 1898 edition raised the limit to about 97 %, and 25 % of the samples, 1901-14, were below this. The limit was again raised in 1914, when it was called "purified," and only 4 % of the samples examined 1916-31 failed to reach it. A few samples bought as "*purified* cream of tartar" are not included.

All the Birmingham samples of 1915-31 were below the B.P. limit of 2 parts of arsenic per million, while 5 % of them were above the B.P. limit of 20 parts of lead per million, containing 24-40 parts. In 16 % the lead was 11-20 parts, and in 79 % it was 0-10 parts. One vendor who sold as "cream of tartar" an article containing 40 parts of lead per million, sold a similar article when asked for "purified cream of tartar." Neither article should contain an excess of lead (cp. Cox, *S.P.A.*, 1924, **49**, 136).

Of the samples examined in England and Wales during 1898-1905, 12.7 % were adulterated; of those 1906-13, 4.4 %, and of those 1920-30, 3.2 %.

Cream of Tartar Substitutes. Most of the adulteration of cream of tartar has been due to the sale, either by intention or accident, of substitutes, "cream powder." The titration of one Birmingham sample before ignition indicated 92.3 % of $\text{KHC}_4\text{H}_4\text{O}_6$, and after ignition only 74.0 %. Acid sulphate of potassium was present, the amount indicated by a determination of (SO_4) being 5.6 %. The calcium present indicated 9.3 % of calcium tartrate. The sulphated ash was equivalent to 101.4 % of $\text{KHC}_4\text{H}_4\text{O}_6$. The same article was first sold as "cream of tartar" and then as "purified cream of tartar."

Particulars of acid phosphate substitutes, and of those adulterated with alum, calcium sulphate, arsenic and lead, are given in subsequent pages (see also White, *S.P.A.*, 1902, **27**, 118).

The most important substitute is acid calcium phosphate, which should be prepared from calcium phosphate and phosphoric acid, when only traces of calcium sulphate will be present. A much

cheaper article may be made by the use of sulphuric acid, and if the theoretical proportions be used, the product would contain 38.4 % of $\text{CaH}_4(\text{PO}_4)_2$ and 61.6 % of CaSO_4 , or one part of the former to 1.6 parts of the latter. Commercial samples contain more or less unaltered $\text{Ca}_3(\text{PO}_4)_2$.

A Report by Hamill (L.G.B. Food Report, No. 13, 1911) stated that there was no difficulty in producing an acid phosphate containing less than 5 % of calcium sulphate, and that a maximum limit of 10 % would provide an ample margin. In the calculation of the percentage present he suggested that all the P_2O_5 should be assumed to be in the form of $\text{Ca}_3(\text{PO}_4)_2$, and that the ratio $\text{SO}_3 : \text{P}_2\text{O}_5$ should not be less than 1 : 7. He considered that the addition of calcium sulphate, which contributed nothing to the value of a baking powder and which might impose an extra and unnecessary burden upon the excretory mechanism, was to be deprecated. In a previous report (No. 8) Hamill quoted the case of cattle, which were fed on rice meal containing calcium sulphate, and either suffered in health or died. The trouble ceased when calcium sulphate was not used.

ANALYSES OF CREAM OF TARTAR SUBSTITUTES (I.)

ANALYTICAL FIGURES	A	B	C	D
As_2O_3 , per million	1	—	3	1
Pb, per million	100	—	—	—
CaO , %	32.6	19.3	17.1	32.7
Al(Fe)PO_4 , %	3.0	9.8	1.6	0.4
P_2O_5 , soluble in water, %	33.5	34.4	—	—
„ insoluble in water, %	15.9	13.9	—	—
„ Total, %	49.4	48.3	30.5	24.5
SO_3 , %	trace	0.8	1.8	28.7
Cl, %	—	—	23.0	0.4
Insoluble in water :				
Loss on ignition, %	0.8	2.6	—	—
Ash, %	35.7	34.6	—	—
Loss in water oven, %	2.6	2.8	—	1.9
Ash, %	82.8	81.7	—	—
Acidity, $\text{KHT} = 100$	71	120	77	61
CALCULATED COMPOSITION OF "ACID CALCIUM PHOSPHATE"				
$\text{CaH}_4(\text{PO}_4)_2$, %	50	69	41	35
$\text{Ca}_3(\text{PO}_4)_2$, %	38	4	11	7
CaSO_4 , %	—	1	3	49
Total, %		74	55	91
% CaSO_4 in it	—	2	6	54
CaSO_4 for 1.0 of $\text{CaH}_4(\text{PO}_4)_2$	—	0.02	0.08	1.4

Sample "A" contained a fair amount (50 %) of acid phosphate, but was contaminated with 100 parts of lead per million. Sample "B" was more acid (69 %); both samples were practically free from calcium sulphate. Sample "C" was weaker, containing 38 % of salt, and a little calcium sulphate. Sample "D" was impure, having

49 % of calcium sulphate. Using Hamill's ratio, and allowance of 10 %, the excess of calcium sulphate above the limit was 1.7 % ($\text{SO}_3 - 1/7 \% \text{P}_2\text{O}_5$) = 43 %.

The following method was used for the calculation of the composition of the above samples, the figures for "D" being given as an example :—

The total CaO was 32.7 %, of which 20.3 % was present as CaSO_4 , and the remainder (12.4 %) combined with 24.5 % of P_2O_5 . The sum (36.9 %) was composed of 33.6 % of CaO, and 66.4 % of P_2O_5 . It was assumed to be a mixture of $\text{Ca}_3(\text{PO}_4)_2$ (containing 54.2 % of CaO) and CaP_2O_6 (containing 28.3 % of CaO). The following formula gave the proportions of the two substances :—

$$\text{Ca}_3(\text{PO}_4)_2 \text{ in the mixture} = \frac{(33.6 - 28.3) 100}{54.2 - 28.3} = 20.4 \%. \quad \text{The}$$

$$\text{percentage of } \text{Ca}_3(\text{PO}_4)_2 \text{ in the powder was } \frac{20.4 \times 36.9}{100} = 7.5 \%, \text{ and}$$

the remainder ($36.9 - 7.5$) = 29.4 % was CaP_2O_6 . Multiplication of the latter by 1.18 gives 34.6 % of $\text{CaH}_4(\text{PO}_4)_2$ in the powder.

If there be any appreciable proportion of $\text{Al}(\text{Fe})\text{PO}_4$ present, the P_2O_5 in it should be subtracted from the total P_2O_5 before the above calculation is made.

The acidity of the samples as compared with cream of tartar was calculated on the assumption, for which there is experimental evidence, that on addition of water CaHPO_4 or Na_2HPO_4 will not decompose NaHCO_3 .

ANALYSES OF CREAM OF TARTAR SUBSTITUTES (II.)

	E	F	G	H	J
As_2O_3 , per million	2	—	1	0	0
Pb, per million	—	—	2	40	50
CaO, %	3.6	—	0	0	trace
Al_2O_3 , %	2.1	—	—	—	—
K_2O , %	15.3	—	—	—	—
Na_2O , %	2.7	—	—	—	—
NH_3 , %	0.2	—	—	—	2.8
P_2O_5 , %	3.1	51.8	38.7	43.5	12.4
SO_3 , %	8.9	—	0	trace	trace
Cl, %	2.2	—	—	—	—
Insoluble in water :					
Loss on ignition, %	—	0	29.5	28.5	67.1
Ash, %	—	0	0	0	0.7
Loss in water oven, %	7.1	0	—	3.8	11.2
„ at 190° C., %	9.5	—	—	—	—
Ash, %	41.3	—	60.0	61.8	3.9
Acidity, $\text{KHT} = 100$	78	142	97	115	33
Starch	—	0	maize	maize	maize

Sample "E" was a complex one, the approximate proportion of constituents being, KHT 57 %, alum 19 %, $\text{Ca}_3\text{P}_2\text{O}_8$ 7 % and salt 4 %. There was little if any tartaric acid present, as a filtered

20 w/v solution containing KCl did not rotate polarised light. Also, there was no charring at 190° C. An experiment showed that tartaric acid lost 24·8 % at that temperature and partly decomposed, while KHT was unaltered.

Sample "F" was 98 % of KH_2PO_4 and 2 % of K_2HPO_4 . "G" contained 74 % of KH_2PO_4 , "H" 68 % of NaH_2PO_4 , and "J" 19 % of AmH_2PO_4 , in each case mixed with maize starch. The part insoluble in water lost a notable amount on ignition. The two last samples contained 40 and 50 parts of lead per million, respectively.

In 1916 the Local Government Board called attention to the presence of arsenic in cream of tartar substitute; one sample contained 643 parts per million, and others 400 parts. There were no large amounts in the Birmingham samples.

ANALYSIS. The figures for the titrations of pure cream of tartar should be practically the same before and after ignition. The determination of phosphoric acid has been previously discussed (p. 73); that of calcium and sulphate may also be necessary. With a superphosphate it is better to determine the soluble and insoluble phosphate separately. Treat 1 gm. with 150 ml. of water overnight. Filter and wash with about 50 ml. of water, to separate the phosphates. If a similar residue in a Gooch crucible be dried and ignited, the loss will probably be an indication of the amount of starch present.

PROSECUTIONS FOR CREAM OF TARTAR. *Stockton.* Sulphate of baryta 17 %, tartrate of lime 8·5 %, sand 1·2 %. Fine 1s. (*Analyst*, 1879, 4, 118).

Forfar. Stucco 30 %, starch 20 %. It was said to be "cream powder." Fine £2 (*B.F.J.*, 1902, 44).

Malmesbury. Lead $\frac{3}{4}$ grain per lb., to the prejudice of the purchaser. Eminent medical evidence was given that such articles were injurious to health, as $\frac{1}{160}$ grain of lead per day, in drinking water, had produced lead poisoning. Although sold by grocers, the magistrates were satisfied that the article was a drug, and fined each of three defendants £5 (*P.J.*, 1903, July 23; *B.F.J.*, 1903, 200).

London, Stratford. Lead at least 4 grains per lb. It was pleaded that the certificate was too vague, as it did not give the constituent parts of the $\frac{3}{4}$ oz. sample analysed. Also, that the impurity had not been added, but "unavoidably" mixed during preparation. Fine £10 (*B.F.J.*, 1904, 21; *P.J.*, 1904, Jan. 23). The vendor subsequently obtained £50 damages from the wholesaler, in the County Court, for breach of warranty (*P.J.*, 1904, June 25).

Loughborough. Arsenic 15 parts per million. Fine £2 (*P.J.*, 1904, March 5).

Melton Mowbray. Arsenic 5 parts per million. It was suggested that the arsenic was due to the vines having absorbed it from the

oils, or having been treated with Scheele's green. Paid costs (*P.J.*, 1904, March 19).

London, Mansion House. Lead 2 grains per lb. Evidence was given that there were two kinds, purified and commercial; also, that it would be necessary to eat 1,200 5-oz. scones to absorb 2 grains of lead. The Lord Mayor, while dismissing the case, recommended that purchasers should be asked which quality they required (*P.J.*, 1904, April 23; *B.F.J.*, 1904, 114).

Hungerford. Lead $\frac{3}{4}$ grain per lb. Each of the four penny packets was labelled "Finest cream of tartar, 98 %." The contents were mixed and divided into three parts. The magistrates dismissed the case, holding that each packet should have been divided. On appeal, *Smith v. Savage* (1905, see p. 21), the magistrates were instructed to convict (*P.J.*, 1904, Oct. 1; 1905, June 24; *B.F.J.*, 1905, 77).

Stoke-on-Trent. Superphosphate of lime 43 %. The sample was taken from a keg which had been supplied to a confectioner as "Warranted pure." The chemical company which sent it was fined £20 (*B.F.J.*, 1906, 193; *P.J.*, 1906, Sept. 8).

Pontypool. Ground rice 45 %. Fine £2 (*P.J.*, 1906, Feb. 21).

Southampton. A mixture of tartaric acid and bicarbonate of soda. Fine 10s. (*Grocer*, 1908, Nov. 21).

Bournemouth. Lead 50 parts per million. The defendant's analyst suggested that only 17 of the 50 parts were lead, the remaining 33 parts being copper. The Government analysts found 25 parts of lead. Fine £1 (*P.J.*, 1917, March 24).

Glasgow. Lead 30 parts per million. The Government analysts found 44 parts. A doctor gave evidence that there was an excess, as he often told a patient to take a teaspoonful every four hours. A professor considered 40 parts per million was just as harmless as 20 parts. Case dismissed (*P.J.*, 1919, Nov. 29; *B.F.J.*, 1919, 113).

Birkenhead. Lead 44 parts per million. Case dismissed, the Bench deciding that lead up to 50 parts per million was safe for ordinary commercial purposes (*Grocer*, 1923, Dec. 22; *Analyst*, 1924, 49, 136; *B.F.J.*, 1924, 5).

Eglington. Acid potassium sulphate 7.9 %, and only 8.3 % of acid potassium tartrate. Fine 10s. (*Grocer*, 1929, Nov. 9).

Lichfield. Maize starch 11 %, phosphate of soda 30 %. The defence was that a substitute had accidentally been mixed with the genuine. Fine £5 (*P.J.* and *Grocer*, 1930, Nov. 1).

PROSECUTIONS FOR PURIFIED CREAM OF TARTAR.

Lambeth. "Lead compounds equivalent to 0.6 grain of lead per lb., to the prejudice of the purchaser, being a food or drug." The alternative "food or drug" in the summons was objected to, but the stipendiary overruled the objection. Fine and costs 17s. (*C. & D.*, 1904, May 21; *B.F.J.*, 1904, 145).

Birmingham. Calcium tartrate and other impurity 16 %. Fine £2 (*P.J.*, 1907, Feb. 23).

PROSECUTION FOR AERATING POWDER. *Ealing.* Calcium sulphate 35 % and starch 31 %. The powder was acid calcium phosphate used for making self-raising flour. For the defence it was stated that the powder was sold as received, except that rice flour was added to make a whiter product. Fine £20 (*P.J.*, 1919, Nov. 15).

ROCHELLE SALT

The 1914 B.P. limit for lead is 20 parts per million, and only two of the sixty-three Birmingham samples exceeded that limit, and that only slightly. In some cases 1 or 2 parts were in the insoluble condition.

LEAD IN ROCHELLE SALT (sixty-three samples)

Parts of lead per million	.	.	0-	6-	11-13	21, 24	Total.
Percentage of samples	.	.	60	30	7	3	100

All the samples were free from arsenic.

ANALYSIS. Ignite 1 gm. at moderate temperature till fuming ceases, add 15 ml. N/2 HCl, and water, and boil. Filter, titrate filtrate back with N/2 NaOH, using methyl orange. One sample titrated in this way indicated 117.6 % $\text{KNaC}_4\text{H}_4\text{O}_6$, 4 H_2O . On titration of the unignited powder with N/2 HCl, using phenol phthalein and boiling, 23 % of NaHCO_3 was shown to be present. Correcting the ignited alkalinity for this addition gave 79.0 % of $\text{KNaC}_4\text{H}_4\text{O}_6$, 4 H_2O , which, added to the 23.0 % of NaHCO_3 gave 102.0 %. Both sodium and potassium were determined, and the former, after correction for the NaHCO_3 , was in less than equivalent proportion to the potassium. It was noticed that small hard lumps were present; these were removed by means of a No. 20 sieve; they weighed 0.6 %, and were cream of tartar.

PROSECUTION. *Birmingham.* Sodium bicarbonate 23 %, and also a small proportion of acid substance, probably cream of tartar. Fine £2 (1924 Report).

CITRIC ACID

The B.P. gives a limit of 20 parts of lead per million; none of the eighty-two Birmingham samples examined 1912-31 reached this limit; 62 % of them had 0-5 parts; 30 %, 6-10 parts; and 8 %, 11-16 parts. Eighty-one samples had less than 1 part of arsenic per million, and a single sample had 8 parts. The B.P. limit for ash is 0.05 %; fifteen samples had 0.002 %, and one had 0.11 %. In three of the eighty-nine samples tartaric acid had been substituted.

Of the samples examined in England and Wales, 1907-13, 2.1 % were adulterated, and 2.0 % of those examined 1920-30.

ANALYSIS. Pratt (*Analyst*, 1912, **37**, 199 ; see also 1917, **42**, 20) and Hartmann and Hillig (*Analyst*, 1927, **52**, 549 ; 1928, **53**, 443 ; 1930, **55**, 396) have published methods for the determination of citric acid in fruits and fruit products.

PROSECUTIONS. *Clerkenwell.* Tartaric acid 100 %. The grocer who sold it was fined £1 (*F. & S.*, 1898, July 9).

Sunderland. Tartaric acid. The oil and colourman who sold it was fined £1 (*Grocer*, 1910, June 25).

Bradford. Lead 90 parts per million. Fine £1, on the defendant promising to destroy the remainder (*P.J.*, 1910, July 2).

London, Old Street. Boric acid 15 %. Fine £1 ; the adulteration was attributed to an accident (*P.J.*, 1914, Jan. 31).

Birmingham. Two vendors were each fined £1 for the sale of tartaric acid as citric acid (1921 Report).

Bristol. Tartaric acid. Fine £1 (*P.J.*, 1931, Feb. 14).

CHAPTER XXVIII

SPICES. SAUCES

Analysis. Mustard. Ginger, facing and washing of root ginger, spent ginger, ground ginger, ginger starch, analysis, prosecutions. Pepper, black pepper, white pepper, pepper compound. Cayenne pepper. Cinnamon, cassia. Mace. Nutmeg. Pimento, allspice. Caraway seed. Mixed spice, pudding spice. Dried thyme. Sauces, curry powder.

Numerous analyses of spices and adulterants examined in the United States have been given by Winton, Ogben and Mitchell (*B.F.J.*, 1899, 238; *Analyst*, 1902, **27**, 54). The United States Department of Agriculture standards for spices are quoted in the *British Food Journal* (1930, 74, 82).

It must not be assumed that identical figures will be obtained from whole spices and their powders. Grinding on a large scale may cause loss of volatile matter, and there may be an increase in the amount of mineral matter in the ground spices.

Of the "spices" examined in England and Wales, 1904-13, 8.3 % were reported adulterated, and 8.5 % of the "spices and condiments" examined 1920-30.

ANALYSIS. The methods for the determination of ash, carbonated ash, insoluble ash, sulphate and carbonate have been previously discussed (pp. 70 ff.). If the ash be high, it should be carbonated and again weighed.

Cold Water Extract. The writer suggested (*B.P. Conf.*, 1896, 359) this determination for ginger; it is also useful for other spices, and for some drugs. Put 1.4 gm. in a small flask, add 70 ml. of water, cork and shake thoroughly, so that no part of the powder is dry. Shake occasionally during the next day, and filter on the third day, and do not wash the residue. Evaporate 50 ml. of the filtrate (= 1 gm. of the powder) to dryness and dry in the water oven two hours or more. With attention to shaking, a shorter time of maceration will suffice, but the above time is safer, as otherwise concordant *incorrect* results may be obtained. The results obtained are not strict percentages, as no account is taken of the dilution by the water present in the powder.

Cold Spirit Extract. Use distilled industrial methylated spirit 64° O.P. instead of water; the writer prefers this to the 90 v/v ordered by the B.P. for ginger.

Ether Extracts. Extract 2 gm. in a Soxhlet with ether for a day, and using another flask for a second day. Distil off most of the

ether, and allow the remainder to evaporate spontaneously. Put the flask in a desiccator (no vacuum required), and weigh daily till constant ; this will give the total extract, and drying in a water oven for a day will drive off the volatile oil.

Essential Oil. For this determination, see Cripps and Brown (*S.P.A.*, 1909, **34**, 519) and Brown (*S.P.A.*, 1910, **35**, 392).

MUSTARD

The proportion of ash in mustard, as deduced from the analysis of samples free from starch and turmeric, is as follows :—

ASH IN MUSTARD (999 samples)

Percentage of ash	. 4.0-	4.1-	4.2-	4.3-	4.4-	4.5-	4.6-	4.7-5.4	Total.
Percentage of samples	2.6	6.1	15.7	22.9	26.5	18.0	7.0	1.2	100

The amount of ash insoluble in HCl rarely exceeds 0.15 %.

OIL (FIXED ETHER EXTRACT) IN MUSTARD (ninety-six samples)

Percentage of oil	. .	29.4-	32-	33-	34-	35-	36-	37 38	Total.
Percentage of samples	. .	2	4	18	30	27	14	5	100

In the following table other analytical figures are given for seven commercial samples passed as genuine, two samples sent with a tender quoting prices, and also for a sample of turmeric :—

ANALYSES OF MUSTARD AND TURMERIC

PERCENTAGES.	Seven Samples.		Samples with		Turmeric.
	Maximum.	Minimum.	I. Tender.	II.	
Loss on drying	. . . 5.7	3.1	—	—	11.4
Ash	. . . 4.66	4.30	4.36	4.58	6.44
Ether extract	. . . —	—	35.5	24.9	7.7
Nitrogen	. . . 5.7	4.9	5.4	6.0	0
Cold water extract	. . . 25.4	21.2	25.1	24.9	12.5
Cold spirit extract	. . . 28.8	16.1	26.6	21.7	—

The range in the cold spirit extract is remarkable, but the other figures have only a moderate range, except the tender sample No. II ; it was one and a half times the price of No. I, and the extraction of part of its oil had made some other figures higher. All the nine samples were free from starch and turmeric.

During the years 1877–1883, 9.4 % of the Birmingham samples were adulterated with starch and turmeric, the average proportion of the adulterants being 28 %. From 1913–1929, only 1.7 % were adulterated, and the average amount of adulteration had fallen to 19 %. In recent years compound mustard has usually been properly labelled as an admixture, and not sold as “mustard.” Wheat flour is usually employed, but potato starch has been detected.

The proportion of adulteration in England and Wales has also shown a steady decrease, as these figures indicate :—

ADULTERATION OF MUSTARD IN ENGLAND AND WALES

Period	1877–	1883–	1894–	1902–13	1919–30
Number of years	6	11	8	12	12
Percentage of adulteration .	17·5	10·7	4·9	3·7	3·4

ANALYSIS. The determination of ash, with testing for starch with iodine and for turmeric with alkali, will usually indicate if a sample of mustard is genuine. Under the microscope, the addition of alkali first makes turmeric cells orange, and then dissolves the colour out of them. Turmeric is generally added to compensate for the paleness caused by the introduction of flour, but in a few instances turmeric alone has been added. One considers this to be undesirable, but the quantity is too small to be called adulteration. When flour is present it should be determined by the microscope, using lycopodium. The proportion may also be approximately determined from the chemical figures as follows :—

$$\text{Percentage of wheat flour and turmeric} = \frac{100(4.3 - \text{ash})}{4.3 - 0.6}, \text{ or}$$

$$\frac{100(35.0 - \text{ether extract})}{35.0 - 1.0}, \text{ or } \frac{100(5.1 - \text{nitrogen})}{5.1 - 1.8}, \text{ or}$$

$$\frac{100(25.0 - \text{cold water extract})}{25.0 - 10.5}.$$

Methods for the determination of the volatile oil have been given by Vuillemin (*Analyst*, 1905, **30**, 59), Luce and Doucet (*Analyst*, 1922, **47**, 353), and Colombier (*Analyst*, 1926, **51**, 308); and for the detection of mustard bran by Leach (*Analyst*, 1905, **30**, 58) and Hertwig and Palmore (*Analyst*, 1924, **49**, 141). Tests for aniline dyes and turmeric have been given by Bohrisch (*Analyst*, 1904, **29**, 372), Schmitz-Dumont (*Analyst*, 1905, **30**, 91), Süß (*Analyst*, 1905, **30**, 308), and Sievers (*Analyst*, 1912, **37**, 557).

PROSECUTIONS. *Birmingham.* A very large quantity of wheat flour and turmeric. Fine 5s. and costs (1873 Report).

Birmingham. Largely wheat flour and turmeric. Prosecution dismissed as it was labelled “an admixture” (1874 Report).

London, Thames. Fixed oil only 19·4 %, instead of at least 30 %. There was no other adulteration. Fine 1s. and 2 guineas costs (*B.F.J.*, 1903, 278).

Dartford. Wheat flour 5 %. Summons dismissed as the analyst had not stated if any change had taken place to interfere with the analysis (*Grocer*, 1905, Jan. 7).

Newcastle-on-Tyne. Wheat flour 30 %. The label on the tin stated: “This mustard compound is a condiment free from any

injurious ingredient." Summons dismissed, and the defendant was allowed 3 guineas costs (*Grocer*, 1909, May 8 ; *B.F.J.*, 1909, 93).

Halesowen. Yellow dye 0.1 %, probably added to colour the mustard. Summons dismissed on proof of warranty (*Grocer*, 1910, Aug. 27).

North London. Wheat flour 32 %, turmeric 1 %. Fine 12s. 6d. and costs (*Grocer*, 1912, June 15).

Woolwich. Foreign starch 15 %. As the Government analysts found only 4 %, the case was dismissed on payment of 2 guineas costs (*Grocer*, 1919, Sept. 27).

Redditch. Foreign starch 5 %. Fine £5 (*Grocer*, 1925, Jan. 17 ; *B.F.J.*, 1925, 18).

London, Marylebone. Wheat flour 10 % and artificially coloured with turmeric. The Government analysts reported the presence of 1 % of turmeric colouring matter, and not more than 0.1 % of wheat starch. Their analytical figures were : volatile oil (vacuum desiccator) 1.36 %, non-volatile oil 29.9 %, fibre 4.1 %, ash 4.5 %, sand 0.1 %. The summons was withdrawn (*Grocer*, 1926, Dec. 18).

GINGER

The *Journal of the Chemical Society* for August, 1917, contains papers by Nomura, Lapworth and others on the pungent principles of ginger (*Analyst*, 1917, **42**, 357-9), a useful summary of which has been given by Grier (*P.J.*, 1917, Oct. 13, Nov. 3, 10). Gingerol is an intensely pungent, inodorous oily liquid, with a high boiling-point. It is soluble in most organic solvents, in weak alcohol and in weak soda solution, but only slightly in water and cold petroleum ether. Zingerone has an extremely pungent ginger taste, and a sweet odour. It occurs in crystals or plates, and is soluble in most organic solvents except petroleum. It is slightly volatile with steam and soluble in dilute alkali, but only sparingly soluble in water. The odour of ginger is not due to these substances, but to the volatile oil (see also Pearson, *B.P. Conf.*, 1919, 420).

The Preservative Regulations do not permit the presence of sulphur dioxide in ground ginger. Of the sixteen Birmingham samples tested for this preservative in 1927, five contained 800 to 3,000 parts per million. These bleached samples were darker than some free from sulphur dioxide. The attention of the local Grocers' Association was called to the adulteration, and the Association brought the question before the Grocers' Federation. There was a great improvement in 1929-31, when six of the ninety-seven different samples examined contained sulphur dioxide, in about the same proportion as before, 120-2,000 parts per million.

During 1892-7, 8 % of the Birmingham samples of ground ginger were condemned because of deficiency of soluble matter, and 1 %

because of addition of mineral matter. During 1912–31, 1·8 % were condemned because of deficiency, and 4·2 % for addition of chalk or sand. In England and Wales, during 1894–9, 4·8 % of the samples of “ginger” were reported adulterated, and 1·9 % of those examined during 1902–13. In 1920–30, 2·2 % of the samples of “ground ginger” were adulterated, while fifty-seven samples of “ginger” examined 1920–1 were all genuine.

Papers on ginger, with analyses, have been given by Young (*S.P.A.*, 1884, **9**, 214); Jones, on the amount of starch (*S.P.A.*, 1886, **11**, 75); Dyer and Gilbard, on exhausted ginger (*S.P.A.*, 1893, **18**, 197), and also Allen and Moor, on the same subject (*S.P.A.*, 1894, **19**, 124); Allen, on extraneous mineral matter (*S.P.A.*, 1894, **19**, 217); Blunt, and other analysts, on spent ginger (*S.P.A.*, 1896, **21**, 309); Clayton (*S.P.A.*, 1899, **24**, 122), Winton, Ogben and Mitchell (*B.J.F.*, 1899, 238), Bevan, Dyer and Hehner, on a spent ginger prosecution (*S.P.A.*, 1899, **24**, 169); Bennett, on pharmaceutical ginger (*P.J.*, 1901, April 27); Liversecge, Bagnall and Lerrigo (*B.P. Conf.*, 1926, 465), and Walmsley (*B.P. Conf.*, 1927, 509).

Root Ginger. The chief varieties of ginger root, or more correctly rhizome, are known as Jamaica, Calicut, Cochin, Japan, and African. Some varieties are imported with the epidermis on, but others, of which Jamaica is an example, have had it removed by scraping. Jamaica ginger is the finest variety, and the only one recognised by the B.P. (see Walmsley, *B.P. Conf.*, 1927, 509). The rhizomes, after careful peeling, are thrown into water, washed, and allowed to remain in water overnight. They are sun-dried for five or six days, again washed, and finally dried in the sun for two days more. Bleaching agents are not used in Jamaica.

The white appearance of some samples of root ginger is due to them having been faced with chalk, lime, or plaster of Paris. It is another example of the craze for whiteness, and is obvious to the purchaser; if the whitewashing is done in moderation it can hardly be called adulteration.

The following are Birmingham analyses of root ginger; the last two were retail samples, the others wholesale:—

ANALYSES OF ROOT GINGERS

PERCENTAGES.	Jamaica.	Cochin.	Calicut.	African.	Washed Japan.	Faced.	
						I.	II.
Ash soluble in water	3·0	2·3	2·7	2·7	1·7	1·5	3·1
Ash insoluble in water, soluble in HCl	0·7	1·7	1·5	1·4	2·2	7·2	3·7
Ash insoluble in HCl (sand)	0·2	0·3	0·3	0·3	0·3	0·3	0·3
Total ash	3·9	4·3	4·5	4·4	4·2	9·0	7·1
Calcium sulphate	—	—	—	—	—	0·8	2·8
Cold water extract	15·1	14·3	12·4	15·0	13·3	10·5	12·7
Ether extract	4·0	4·5	—	6·1	5·0	—	—
Methylated spirit extract after ether	3·0	3·0	—	3·9	7·0	—	—

The Jamaica contained the smallest ether extract, and was much lower than the African. The large amount of ash insoluble in water but soluble in HCl in washed sample No. I. (7.2 %) was chiefly due to chalk, as the calcium sulphate was only 0.8 %, while the 3.7 % in No. II. was chiefly due to calcium sulphate (plaster of Paris). Four other samples of faced ginger had less than 6.0 % of ash.

Washing and lime-washing ginger is a special trade, and most ginger that is sold is washed at least once in this country, in addition to any washing before exportation. Washing can be properly done without any appreciable exhaustion, but if the ginger is allowed to soak for days the vendor will be liable, under sect. 5 of the 1928 Act, for selling as ginger an article part of which has been abstracted "so as to affect injuriously its nature, substance or quality" without making disclosure. The defence that such an article has been sold as imported is obviously unsatisfactory, as the purchaser is equally prejudiced wherever the abstraction has been made (see *S.P.A.*, 1896, 21, 313; 1899, 24, 124, 169). The rather small amounts of ash soluble in water (1.5 %), and cold water extract (10.5 %), in faced sample No. 1. above indicate an undue amount of washing during the facing process. A sample of Calicut ginger, not included in the above average, containing 1.4 % of ash soluble in water and only 7.0 % of cold water extract had been badly treated before exportation.

An experiment was made on the effect of soaking a "washed Cochin root ginger" for various periods in tap water:—

EFFECT OF SOAKING ROOT GINGER

Time of soaking	None.	3 hours.	6 hours.	1 day.	4 days.
Ash soluble in water, %	2.8	1.9	1.5	1.0	0.9
Cold water extract, %	11.2	9.8	9.0	7.7	6.4
Ash of cold water extract, %	3.2	2.5	2.0	1.3	—

The proportion of water to ginger was probably considerably greater than would be necessary in commercial practice, but it is evident that washing must be done with care.

Spent Ginger. Besides samples of ginger which have been deficient through carelessness, there have been samples on the market from which the flavouring ingredients have been intentionally extracted by water or spirit. The writer has made experiments on the effect of solvents on commercial ground ginger (*B.P. Conf.*, 1896, 359). The ground ginger was soaked with occasional shaking for seven days in about four times its weight of the solvent: the results are calculated to the same percentage of water as in the original powder; the two last in the table are commercial samples of "spent" ginger:—

EFFECT OF SOLVENTS ON GROUND GINGER

Solvent:— PERCENTAGES.	None.	Rectified Spirit.	Proof Spirit.	Water.	"Spent,"	
					I.	II.
Ash soluble in water	2.4	2.2	1.7	1.0	0.2	2.2
Total ash	4.9	5.0	4.3	3.3	1.4	5.0
Cold water extract	11.8	10.5	6.8	4.7	8.4	11.2
Cold methylated spirit extract .	6.5	2.9	4.5	5.6	2.2	1.7
Ether extract	5.5	1.8	3.8	5.4	2.4	1.9
Methylated spirit extract after ether	4.6	2.3	2.3	3.2	1.1	2.1

If judged by the ash soluble in water and cold water extract only, the sample extracted with rectified spirit, and spent ginger No. II., would be passed as genuine, but they are condemned by the low ether and methylated spirit extracts. On the other hand, the water-extracted sample has a satisfactory cold methylated spirit extract.

The ratio between the ash soluble in water and the cold water extract varies considerably in various samples of spent ginger, and the writer has used a composite standard of the sum of the two, which should not be less than 12 %.

In giving a certificate for deficient ginger it is better to give the percentage of soluble constituents *deficient*, rather than to suggest the *addition* of spent or exhausted ginger. It is much easier to prove a deficiency than an addition, and, on the other hand, the defence may prove that there has been no addition.

Ground Ginger. Three samples of ground Jamaica ginger, from Birmingham wholesalers, yielded 3.7–3.9 % of total ash, with 2.6–2.8 % soluble in water; the cold water extract was 17.1–18.2 %, and 7.0–7.6 % of 90 v/v alcohol extract. These figures are much better than the B.P. limits.

The following ranges of ash constituents are calculated from analyses of samples bought from retailers; all samples are included, and extreme figures are due to samples which have been partly exhausted or adulterated with chalk or sand:—

ASH ANALYSES OF GROUND GINGER, 1892–1929 (500 samples)

Ash soluble in water, % .	1.6–	1.8–	2.2–	2.6–	3.0–	3.4–4.4	Total.	
Percentage of samples .	8	16	39	23	11	3	100	
Ash insoluble in water, soluble in HCl, % .	0.7–	1.0–	1.4–	1.8–	2.2–	2.4–	3.0–13.5	Total.
Percentage of samples .	8	10	17	22	17	13	13	100
Ash insoluble in HCl (sand), % .	0–	0.2–	0.4–	0.6–	0.8–	1.0–	1.2–4.2	Total.
Percentage of samples .	7	25	30	17	7	7	7	100
Total ash, % .	2.2–	3.0–	4.0–	5.0–	6.0–	7.0–	8.0–16.4	Total.
Percentage of samples .	2	16	28	25	21	4	4	100

The above figures indicate that the percentage of ash soluble in water should not be less than 1.8 %. The ash insoluble in water but soluble in acid includes, in addition to that natural to ginger, any

chalk, and also sulphate of lime, whether due to coating or to treatment with sulphur dioxide. Allowing for such additions, this figure should probably not exceed 2.4 %. The sandy matter on the rhizomes can evidently be removed by washing, and should not exceed 0.7 %.

The ranges of the corresponding figures for extracts are given below ; adulterated samples are again included. The water figures depend on over 500 samples and the others about half as many :—

EXTRACTS FROM GROUND GINGER BY SOLVENTS

COLD WATER EXTRACT, %	5.9-	9.0-	10.0-	11.0-	12.0-	13.0-	14.0-	18.4	Total.
Percentage of samples	6	3	12	19	23	20	17		100
METHYLATED SPIRIT EXTRACT, %	3.1-	5.0-	5.4-	5.8-	6.2-	6.6-	6.8-	8.4	Total.
Percentage of samples	9	15	15	13	15	14	19		100
ALCOHOL 90 v/v EXTRACT, %	4.1-	5.6-	6.0-	6.4-	6.8-	7.2-	9.5		Total.
Percentage of samples	13	21	13	19	15	19			100

In nearly two-thirds of the samples the cold water extract was between 11 % and 14 %. In both sets of spirits figures the percentage of samples was much more uniform over the range of composition. In a few samples the ash soluble in water was compared with the ash of the cold water extract. The average of the former was 2.7 %, and of the latter 3.4 %.

MOISTURE IN GROUND GINGER, 1922-9 (150 samples)

Water, %	9.0-	10.0-	11.0-	12.0-	13.0-	13.5	Total.
Percentage of samples	12	38	26	19	5		100

A large proportion of the samples contained between 10 % and 13 % of water.

In connection with the analysis of Gregory's powder, the amount of ginger insoluble in E/2 acetic acid was determined. About one-quarter of the samples was found to be included within each of the following limits :—76.0-, 77.0-, 78.0-, and 79.0-80.4. The following formulæ were also found to be approximately correct :—

$$\begin{aligned}\text{Percentage of ginger} &= 1.19 (100 - \% \text{ moisture} - \% \text{ ash}) \\ \text{,,} \quad \text{,,} &= 1.05 (\% \text{ cold water extract} + \% \text{ insoluble in acetic acid}).\end{aligned}$$

Ginger Starch. The starch grains are usually ovate, but a number are round (28 % in one sample), most of them have one or more flat sides ; many are pointed at one end. The hilum is very faint, and only a faint cross is given with polarised light. Like wheat starch, the grains are flat. The length of the grains varies from 7μ to 42μ , but very few grains are over 40μ . The proportion of grains of different lengths was as follows :— 7μ -, 22 % ; 16μ -, 54 % ;

and 24μ —, 24 %. The average length of 200 grains was 21μ and the average breadth 16μ . Wheat starch is a possible adulterant, but this starch has a number of grains 40 – 50μ , and some grains are 76μ ; very few of them are pointed.

The grains of Japan ginger are very different to other varieties. According to Kimura and Watanabe (*Q.J.P.*, 1929, 342), large partially compound grains consisting of two to six partial grains, and small compound grains, are common. The single grains are round, 10 – 25μ , or ovoid, 20 – 40μ long.

ANALYSIS. For the detection of exhausted ginger, Dyer and Gilbard (*loc. cit.*) emphasised the value of the determination of the alcoholic extract after ether extraction. They found the volatile oil to be too variable to be of much use. The determination may be made by extracting 5 gm. in a Soxhlet with ether for one day, then, using a sand bath, with methylated spirit for two days.

The writer suggested the determination of cold extracts (see p. 409). The B.P. 1914 directs the use of a 5 w/v mixture (instead of 2 w/v above) and twenty-four hours only. With water there is little difference between the two methods, but with 90 v/v alcohol, the B.P. method gave an extract of only 5.3 %, against 7.3 % by the other method.

If the water extract be repeatedly evaporated with water to remove any volatile oil, the remaining extract has a pungent taste. This is an answer to the statement sometimes made—that the extract consists merely of flavourless ingredients, and is therefore no measure of the quality of the ginger.

Garnett and Grier (*B.P. Conf.*, 1907, 443) have described a test for cayenne in ginger. Heat a tincture of the ginger with a little caustic alkali for fifteen minutes. Evaporate off alcohol and acidify with HCl. Shake out with ether in a test tube. If cayenne be present, the ether solution will have a pungent, biting taste, while ginger yields no pungency. See also La Wall (*Analyst*, 1909, **34**, 321).

PROSECUTIONS FOR GROUND GINGER. *Sheffield.* Exhausted ginger 40 %. For the defence it was argued that there was no standard of pure ginger before the Court, and that it was no offence if the purchaser got something weaker than he expected. The prosecution replied that there could not be a better standard than pure ginger, and that was where the analyst started. The magistrates, considering that there had been carelessness but no intentional fraud, fined the defendant 10s. (*C. & D.*, also *F. & S.*, 1894, Jan. 27).

Birmingham. Exhausted ginger 75 %. Fine 10s. and costs (*F. & S.*, 1894, Feb. 3).

London, Marlborough Street. Spent ginger 70 %. It was stated that the spice grinders held that the admixture of spent ginger with a high quality of other ginger was not adulteration. The magistrates

decided that taking action under sect. 6 (prejudice to the purchaser) was incorrect, but permitted the summons to be amended and fined the vendor 10s. under sect. 9 (abstraction) (*F. & S.*, 1894, Aug. 11).

Bingley. Sand and extraneous mineral matter not less than 5 %. The defendant's analyst had found 2.5 % of sandy matter, but did not look for chalk. The Government analysts reported 8.82 % of mineral matter, of which 2.11 % was sand. They did not consider the quantity excessive for unwashed ginger. The magistrates dismissed the case, considering that if people wanted cheap ginger they must expect to have a certain amount of dirt in it (*F. & S.*, 1894, Aug. 25; see also *Analyst*, 1894, **19**, 217).

Cardiff. Spent or exhausted ginger 25 %. There were two summonses, one for the sale of a *food*, from which part had been abstracted, and one for the sale of a *drug* to the prejudice of the purchaser. The magistrates could not come to a decision and dismissed both cases (*F. & S.*, 1894, Sept. 29).

Halifax. Sulphate of lime, 10 %, which the Public Analyst considered was due to the addition of ground gypsum. The defendant's analyst attributed it to sulphite of lime used in bleaching the ginger, and which had settled at the bottom of the tin. Fine £1 and costs (*C. & D.*, also *F. & S.*, 1894, Oct. 20).

Birmingham. Extraneous mineral matter 5 %. The Bench considered the case so trivial that they could not convict. (The sample contained 11.9 % of ash, of which 4.2 % was sand (*F. & S.*, 1894, Dec. 8).

Liverpool. Chalk and sand 12 %. Fine 10s. (*F. & S.*, 1895, March 16).

Haslingden. Spent ginger 25 %. The defendant suggested that it had been affected by being kept in a damp place. Fine 10s. (*F. & S.*, 1897, May 29).

Cranbrook. Exhausted ginger 50 %. The sample was stated to have 0.8 % of soluble ash, and between 4 % and 5 % of matter soluble in water. It was said to have been made from washed Cochin ginger. Fine £5 (*F. & S.*, 1898, Nov. 19; *C. & D.*, 1898, 53, 821, 861).

Barnsley. Ginger exhausted of its pungent principles, at least 50 %. The report of the Government analysts indicated: soluble in 90 % alcohol 4.56 %, volatile oil 0.43 %, fixed oil and resin 4.43 %, soluble in alcohol after ether 1.08 %, soluble in water after alcohol and ether 3.70 %—total ash 2.33 %, ash soluble in water 1.03 %. The opinion was expressed that the sample in question was a washed Japan or similar low-grade ginger. Several public analysts gave evidence that these figures corroborated the adulteration. Case dismissed (*C. & D.*, 1899, April 15; *B.F.J.*, 1899, 152; *Analyst*, 1899, **24**, 169).

Ripley. Pea flour 100 %. It was stated that an assistant had put pea flour in a drawer intended for ground ginger. Costs paid (*B.F.J.*, 1902, 251).

Church. Calcium carbonate 2 %, and barium sulphate 4.5 %. The magistrates did not consider the vendor was to blame, and the case was withdrawn as he promised to destroy the article (*B.F.J.*, 1903, 185 ; *P.J.*, 1903, Aug. 1).

Birmingham. Ground chalk 9 %. The defendant said that the ginger was guaranteed pure to him, and he thought the stem ginger had been limed. As the amount of lime was so large, the Bench concluded the article had been adulterated, and fined the defendant £1 (*Grocer*, 1915, June 12 ; *B.F.J.*, 1915, 137).

Birmingham. Ground chalk 9 %. The sample was bought from the same shop as the sample just mentioned. The vendor said he had recently bought the business and that the ginger was part of the stock. He undertook to destroy the remainder, and was only ordered to pay 4s. costs (*Grocer*, 1920, Nov. 27 ; *B.F.J.*, 1920, 115).

Southport. Applying a false trade description, "Ground ginger," to exhausted or spent ginger. Defendant said he bought root ginger at salvage sales, and also bankrupt stock, and had it ground. He had no idea it was wrong. Fine £5 (*B.F.J.*, 1916, 276).

Birmingham. About 30 % of certain soluble constituents removed by treatment with water. The Public Analyst stated that the ash soluble in water was 1.6 %, and the cold water extract 6.9 %, making a total of 8.5 %, and that genuine ginger should contain a total of 12 %. Evidence was given for the defence that Calicut ginger had been imported and ground as received, without washing in England. The defendant's analyst, while agreeing with the analysis, said the ginger was a new variety, some samples of which were very low in water-soluble extract. The magistrates dismissed the case, thinking that, in spite of the low figures, the sample was genuine (*Grocer*, 1916, July 15).

Mold. Sand 27 %. The sample was taken from a new tin, and the vendor was ordered to pay costs (*P.J.*, 1917, May 18).

Blackburn. Essential oil deficient, 0.74 % being present, instead of 1.2-2.0 % in a genuine sample. Notice of warranty was given too late. Ordered to pay costs (*B.F.J.*, 1919, 58).

Birmingham. About 30 % of certain soluble constituents had been removed by treatment with water. The ash soluble in water was 1.8 % and the cold water extract 6.6 %. Each of the two partners was fined 5s. (1924 Report).

London, West Ham. Sulphur dioxide, 1,564 parts per million. The defence stated that the humid atmosphere of India and Cochin required the use of preservative in drying the roots. They were bleached with lime, which retained the sulphur dioxide. Ordered

to pay £1 costs (*Grocer*, 1929, May 25, June 1; *Analyst*, 1929, **54**, 419; *B.F.J.*, 1929, 79).

London, Thames. Sulphur dioxide 0.08 %. Fine £2 and 10 guineas costs (*B.F.J.*, 1931, 58; *Grocer*, 1931, April 25, May 2).*

PROSECUTIONS FOR ROOT GINGER. *Birmingham.* Contained 25 % less of certain essential constituents than genuine ginger of lowest quality. The ash soluble in water was 1.2 %, and the cold water extract 7.6 %. Fine 5s. (1897 Report).

Cupar. Sulphur dioxide. Fine 20s. (*Grocer*, 1930, Feb. 1).

PEPPER

There are various kinds of pepper sold: black pepper, white pepper, long pepper, "pepper," pepper compound, cayenne pepper, and cubeb, or tailed pepper; the two latter are not derived from the genus "Piper."

Black Pepper. This was described by the 1898 B.P. as "The dried unripe fruit of *Piper nigrum*." Detailed analyses have been published by Heisch (*S.P.A.*, 1886, **11**, 186), who had been informed by a large grinder that the ash should not exceed 6 %; by Johnstone (*S.P.A.*, 1889, **14**, 41), none of whose nine samples contained 5 % of ash; also by Gladhill (*Analyst*, 1904, **29**, 117). Hartel and Will give figures (*Analyst*, 1908, **33**, 18), and suggest 6.5 % of ash as a limit. Two series of experiments on grading black pepper have been described by Trillich (*Analyst*, 1891, **16**, 235).

ASH IN GROUND BLACK PEPPER (ninety-four samples)

Percentage of total ash	.	1.8-	5-	6-	7-	8-13.1	Total.
Percentage of samples, 1875-87	.	11	13	20	13	43	100
" " " 1894-1927	.	33	33	14	20	0	100

In the earlier period, a large proportion of the samples contained too much mineral matter, either by wilful addition, or by failure to clean the berries. The high ash in one of the few samples of the latter period was due to the use of a rusty mill, and another had 1.1 % of chalk present; 6.5 % was the limit used for total ash. The ash in eleven samples of unground pepper-corns varied 2.8-5.7 %, and the ash insoluble in acid did not exceed 0.5 %. Tested by Hepburn's method, black and white pepper-corns each yielded CO₂ equal to about 0.2 % of chalk. The amount of crude fibre in the black corns varied 10.9-14.0 %.

The extract dissolved by cold methylated spirit from seven samples of black pepper and corns varied 7.8-9.9 %; the cold water extract of fifteen samples showed greater variation, 5.1-10.8 %, though twelve of them were over 7.8 %. The fixed ether extract, 7.2-9.1 %, was similar to white pepper, but the volatile, 0.8-1.1 %, was higher.

* See Addenda, p. 278.

White Pepper. This is obtained from the same vine as black pepper, but in this case the berries are allowed to ripen more, and the hull removed either by machinery, or by rubbing with hands or feet after soaking in water till the outside is soft. There is a considerable variation in the white pepper of commerce; some, the more expensive, is pale in colour and has only 0.6 % of ash; other darker samples may have twice or thrice as much ash. The difference depends on grading and sieving, and also on how many of the coats of the berry have been removed before grinding.

A number of analyses dealing with the grading, facing and bleaching of white pepper have been given by Stock (*S.P.A.*, 1891, 16, 224; 1892, 17, 34). He considered that the ash of white pepper should not exceed 2.75 %. The previously mentioned papers of Heisch, Johnstone, Stock and Gladhill also deal with white pepper.

TOTAL ASH IN WHITE PEPPER (2,000 samples)

Percentage of total ash	0.3-	0.6-	1.0-	1.4-	1.8-	2.2-	2.6-	Total.	Maximum.
Percentage of samples:									
1876-95	.	0	8	43	17	15	6	11	10.4
1896-1915	.	1	22	45	18	9	3	2	100
1916-29	.	4	33	42	15	4	1	1	100

In the first period there was gross adulteration with mineral matter, but there has been a great improvement, and there is nothing unreasonable in expecting white pepper to have less than 2 % of total ash. It is rarely that the ash insoluble in acid exceeds a trace. None of the twenty-six samples examined in 1928 indicated the presence of sulphur dioxide.

The fixed ether extract in twelve samples varied from 6.9-9.2 %, and the volatile 0.2-0.5 %. The cold methylated spirit extract in eleven samples varied from 7.6-9.8 %.

COLD WATER EXTRACT IN WHITE PEPPER (forty-nine samples)

Percentage of cold water extract	0.8-	1.5-	2.5-	3.5-5.1	Total.
Percentage of samples	.	4	61	27	8
					100

The question of the permissible amount of husk is a difficult one; apparently it cannot be entirely removed, and, as a certain amount of grading appears to be legitimate, there must be some concentration of fibre in the darker grades. In twenty samples of white pepper examined in Birmingham the crude fibre varied 0.1-5.6 %; the only samples higher were adulterated with 5 % of mineral matter. Published analyses of pepper husk give from 21-32 % of crude fibre. If 28 % be taken as a mean figure, multiplication of fibre by 3.5 will give husk. If, also, 6 % be taken as a liberal allowance for fibre in white pepper, the adulteration may be calculated as follows:—

$$\text{Husk in excess, \%} = \frac{(\text{Crude fibre \%} - 6) 100}{28 - 6}$$

$$\text{or } (\text{Crude fibre \%} - 6) 4.5.$$

Consult papers on the subject by Heisch (*S.P.A.*, 1888, **13**, 149), Stokes (*S.P.A.*, 1887, **12**, 147), Smith, Alfend and Mitchell (*Analyst*, 1926, **51**, 584), and also the reports of prosecutions, 1911–1914, mentioned below.

The practice of tinting white pepper with turmeric has been adversely commented on by Tankard (*Analyst*, 1930, **55**, 631).

In addition to mineral matter, Birmingham samples have been adulterated with starch, poivrette, long pepper, and 25 % of ground ginger. There has been a great decrease in the amount of adulteration; during 1876–95, 8.3 % of the samples of white pepper were adulterated; during 1896–1915, adulteration fell to 0.6 % and in 1916–29 to 0.3 %.

Pepper. The standards for white pepper should not be applied to an article sold as “pepper,” which, the writer considers, may contain black, white or long pepper, all being obtained from the genus “*Piper*.” This is not always appreciated; a packer received a guarantee for an article as “Genuine pepper,” but packed it and labelled it as “Genuine *white pepper*.”

Pepper should not contain added husk, and therefore the fibre should be lower than that of black pepper. It is usually darker than white pepper and contains a little more ash. Of the twenty-three samples bought as “pepper,” 1916–30, 42 % contained 0.6–2.0 % of ash, 29 % from 2.0–4.0 %, and 9 %, 4.0–4.9 % of ash.

According to the L.G.B. Report for 1886, 13 % of the samples examined in England and Wales classified as “pepper” were adulterated. The proportion gradually fell to 1.6 % in 1893, the average for the period being 6.7 %; during 1894–1903, it was 1.6 %; during 1904–13, it was 0.9 %, and during 1919–1930, 0.6 %.

MICROSCOPICAL EXAMINATION. The variation in diameter of the starch grains of white pepper has been found to be as follows :—

MEASUREMENTS OF PEPPER STARCH

Diameter of starch grains, μ	. 1.6–	3.0–	4.0–5.0	5.4	Total.
Percentage of samples . . .	45	33	21	1	100

Rice starch is nearest in size to pepper starch, but as about three-quarters of those grains exceed 5μ , against 1 % of pepper, if the lenses of the microscope are so arranged that one micrometer eyepiece division equals 5μ , grains of rice will easily be detected.

Wallis (*S.P.A.*, 1915, **40**, 190) has described structures in the apex of white pepper berries and given drawings.

Before 1884, someone discovered that ground olive stones resembled certain structures in pepper, and a large quantity was imported from Italy, under the name “poivrette,” for “blending” with pepper. Campbell Brown exposed this fraud and described the appearance of the adulterant (*S.P.A.*, 1887, **12**, 23, 47, 72); see also the Paris Municipal Laboratory test (*Analyst*, 1890, **15**, 119).

The microscopical structure of long pepper as compared with white pepper has been given by Rimmington (*S.P.A.*, 1888, 13, 81), with drawings.

PROSECUTIONS FOR BLACK PEPPER. *Glasgow.* Added starch 20 %, or thereby. Fine 10s. (*Analyst*, 1884, 9, 71).

Glasgow. Sand 3 %, mineral matter 4 %, pepper husk 7 %, in excess. The mineral matter amounted to about 16 %, instead of 7 %, and sand to about 6 %, the Government analysts' limit being 3½ %. Fine 15s. (*Analyst*, 1889, 14, 59).

London, Mansion House. Common salt 16 %. Fine and costs 27s. (*B.F.J.*, 1901, 301).

Stonehaven. Foreign starch 15 %; the Government analysts confirmed the result. Fine £3 (*B.F.J.*, 1903, 136).

PROSECUTIONS FOR WHITE PEPPER. *Birmingham.* Rice 25 %. Two vendors each paid 2s. 6d. fine (1887 Report).

Liverpool. French chalk. Fine £1 (*B.F.J.*, 1900, 264).

Liverpool. Bleached pepper husks 7 %. The sample had been dyed yellow. The Public Analyst stated that only forty-nine of 522 samples examined contained any bleached husk. The grinder of the pepper admitted the sample had been dyed. The stipendiary dismissed the case in the absence of a standard (*B.F.J.*, 1902, 230).

Blackburn. Woody tissue resembling ground olive stones 10 %. The adulteration was stated to produce £10 per ton extra profit. Fine £20 (*B.F.J.*, 1903, 69).

Colne. Bleached husks at least 10 %. Fine £5. The vendor appealed to Quarter Sessions against the conviction. One of the public analysts gave evidence that the husks had been bleached with chloride of lime and sulphuric acid. One of the Government analysts found that it contained about 10 % of added bleached pepper husk. Other public analysts gave evidence as to the absence of bleaching. The grinder of the pepper said the trade demanded "a speck" and 10 % of black pepper husks were added. The Chairman said the addition of 10 % of husk was admitted, and as the Court found that this 10 % was bleached, the appeal would be dismissed with costs (*Grocer*, 1903, Nov. 28; *B.F.J.*, 1903, 204, 272).

Liverpool. Ground rice 50 %, bleached pepper husks 10 %, added to produce the appearance of normal pepper. Fine £2 (*Grocer*, 1904, Dec. 3).

London, Tower Bridge. Pepper husk 10 % in excess of the 8 % allowed. The Public Analyst stated that he had examined white peppers, and found 6 % of husk in Penang, 5.5 % in Siam, and 5.2 % in Singapore. An analyst for the defence found 7.9 % of fibre, equivalent to 9 % of pericarp; he considered it pure, and had found 1.2 % to 14.4 % of husk in commercially pure white pepper. The magistrate said it was a low grade of white pepper just on the

border line, and dismissed the summons, allowing 5 guineas costs (*Grocer*, 1911, May 27 ; *B.F.J.*, 1911, 120).

Blackburn. Pepper husks, not less than 10 %. The Public Analyst said about 12.5 % was present, but he had allowed 2 % as inevitable. He had found 7.83 % of fibre, 2.6 % ash, 1.1 % essential oil, and 6.2 % piperin. He defined "white pepper" as black berries deprived of the whole of the husk—about six outer skins. A spice grinder stated that in white pepper there should be no husk, but Penang contained up to 2.5 %, other varieties less. A Government analyst said his standard for crude fibre was 6.17 %, and that the 7.57 % present represented at least 5 % excess husk. An analyst for the defence defined white pepper as black pepper-corns deprived of as much of the dark layers as to render them white when ground. A public analyst stated he would pass as genuine a sample containing 9 % of crude fibre. The magistrates considered that the evidence proved that the husk is greatly inferior in value to genuine white pepper, and accordingly, if deliberately added, or through insufficient decorticating, or by process of sieving, it is allowed to remain in the genuine pepper in undue quantity, it would constitute adulteration. Fine £1. An appeal to Quarter Sessions followed. A Government analyst included all six layers as husk which should be excluded. An analyst for the vendors described the three outer layers as "black husk," and said that the next three layers were the richest of the whole pepper, and might be included in white pepper. A representative of the grinders stated that it was a third or darkest grade, obtained from Singapore white pepper, and that the concentration of husk in it was due to grading. It was argued (1) that pepper husks were a normal ingredient, (2) that there was no standard for the amount of husk, and (3) that it was impossible to determine with anything like certainty the amount of black husk present. The Recorder allowed the appeal ; the evidence failed to prove what quantity of husk was present, and did not satisfy him that the quantity was so considerable to prevent it being properly described as white pepper (*Grocer*, 1913, Nov. 15, Dec. 27 ; 1914, April 11, 18, July 4 ; *B.F.J.*, 1914, 19, 40, 68, 125).

Liverpool. Black pepper husks, at least 15 %, some of which had been partially bleached. The Public Analyst stated that the sample contained 9.36 % of woody fibre, but he had never found more than 3.9 % in samples he had ground himself. Trade evidence was given that white pepper should be derived from corns with the outer husk removed. A director of the defendant firm denied that any bleaching or addition of husk had been done at their mills, but that the sample was graded Singapore and Penang pepper. A public analyst for the defence said there should be close on 7.15 % of fibre, which gave a figure of about 6 % pepper husks. Subsequently the Government analysts reported the presence of 9.33 % of crude fibre,

corresponding to about 14 % of excess husk. The stipendiary was satisfied that white pepper was black pepper from which the whole of the husk had been removed ; with an allowance of 2 % or 3 %, it was evident a large excess of husk was present in the sample. Fine £10, and the same fine in two other cases when not less than 10 % and 8 % respectively, of black pepper husks were present (*Grocer*, 1914, Feb. 28, March 4, April 4 ; *B.F.J.*, 1914, 95, 139).

Upton-on-Severn. Common salt in excess 4 %, allowing for 0.5% which should not be exceeded in genuine pepper. Ordered to pay costs (*Grocer*, 1917, Oct. 27 ; *B.F.J.*, 1917, 215).

Barnsley. Pepper husks 20 %. 15 % of fibre husks had been allowed for genuine pepper, which was a high maximum. Paid costs. (*Grocer*, 1924, July 5 ; *B.F.J.*, 1924, 78).

Birkenhead. Rice starch 75 %. The wholesaler had described it as "Perfectly prepared white pepper," but on correspondence admitted that it was a "Pepper compound," only to be sold with a label. Fine 6s. (*Grocer*, 1930, March 8 ; *B.F.J.*, 1930, 40).

PROSECUTIONS FOR "PEPPER." (Some of the samples may have been bought as "white pepper.")

Lambeth. Rice flour 50 %. Fine £1 (*F. & S.*, 1893, May 6).

Manchester. Clay 10 %. Fine 2s. 6d. (*F. & S.*, 1898, Nov. 5).

Dewsbury. Sulphate of lime. Fine £1 (*B.F.J.*, 1900, 263).

Lambeth. Ground maize 40 %. The case against the retailer was dismissed, but the wholesale dealer was fined £20 (*B.F.J.*, 1900, 296).

Raustenstall. Ground olive stones 10 %. Fine £20 (*B.F.J.*, 1903, 116).

Clerkenwell. Lead chromate 0.3 %, rice 60 %. Fine £4 (*P.J.*, 1904, Dec. 10).

Sheffield. Obtaining money by false pretences by selling an article containing 50 % of ground rice as pure pepper. Vendor sent to prison for four months with hard labour (*Grocer*, 1912, June 29 ; *B.F.J.*, 1912, 118).

Clonmacate. Material having the characteristic of rice starch 50 %. The label stated that it was a mixed article containing various spices and ingredients added during grinding, and was warranted to be of choice quality. Fine and costs, 15s. The Chairman described it as a deliberate fraud, and wished he had been able to inflict the maximum penalty on the wholesale firm who supplied the retailer (*Grocer*, 1928, Sept. 15 ; *B.F.J.*, 1928, 109).

Portsmouth. Starch foreign to genuine pepper 45 %. The article, described as "Kashmere pepper," was taken in course of delivery, but the prosecution was dismissed as the retailer had ordered "as per sample," when he did not think the sample was genuine pepper (*Grocer*, 1929, March 9 ; *B.F.J.*, 1929, 48).

Derry. Material having the characteristics of bean flour about 50 %. Fine 50s. and costs (*Grocer*, 1929, July 6).

London, South-Western. Rice and maize starches 42.5 %. As it was a first offence, 2 guineas costs only was ordered to be paid (*Grocer*, 1930, Feb. 22 ; *B.F.J.*, 1930, 28).

Enniskillen. Turmeric 10 %. Another analyst found only 1 %, and the Government chemists reported 1.1 % of turmeric to be present. An analyst gave evidence that the public liked pepper tinted with 0.25–1.0 % of turmeric, and the manager of a pepper factory, turning out 20–25 tons of pepper per week, stated that 75–80 % of their pepper was tinged with turmeric. The magistrates decided that the turmeric was purely a colouring matter, and that the quantity present was negligible, and that white pepper containing turmeric was sold more than other pepper, and they dismissed the case, allowing the defendant 2 guineas costs. This decision covered a second case in which the Public Analyst certified 5 % of turmeric, and the Government chemists 0.2 % (*B.F.J.*, 1930, 89, 110 ; *Grocer*, 1930, Aug. 30, Oct. 4).

PROSECUTION FOR PEPPER COMPOUND. *Sheffield.* Ground rice 50 %. The defendant proved a warranty and the case was dismissed. Proceedings were afterwards taken against the wholesaler for giving a false warranty. The Public Analyst stated that the quantity of starch was excessive, but he could not fix a standard. An analyst for the defence also said that there was no standard, and that the rice flour had been impregnated with capsicum, which was 500 to 1,000 times as pungent as pepper, and also other ingredients. The case was dismissed (*Grocer*, 1929, Aug. 24, Oct. 19 ; *B.F.J.* 1929, 119 ; *Analyst*, 1929, **54**, 663).

CAYENNE PEPPER

The adulteration of this spice in England appears to be uncommon, twenty-three samples examined in Birmingham, 1916–30, all being genuine. Nicoloff (*Analyst*, 1924, **49**, 394) has recorded an outbreak of lead poisoning in Bulgaria due to 20 % of red lead in cayenne.

The B.P. (1914) gives a limit of 7 % of ash for the fruit. The figures of nine samples given by Lenton varied 4.7–5.8 % (*P.J.*, 1901, Nov. 16).

ASH IN CAYENNE PEPPER (thirty-eight samples)

Percentage of ash . . .	4.4–	5.0–	6.0–	7.1, 8.2	Total.
Percentage of samples . .	16	32	44	8	100

The amount of ash insoluble in 3E.HCl was determined in twenty-six samples. It was 0.08–0.4 % in 54 % of them, 0.5–0.8 % in 31 %, and 1.0–1.2 % in 15 % of them. In five samples the lead was 4–7 parts per million, and in six it was 8–13 parts. Twelve samples had 13.6–17.3 % of non-volatile ether extract, and three others 18.8–21.2 % ; the usual B.-R. 40° of the fat was 60–62

(R.I. 1-4659-72), but extremes were 57 and 65. Three samples gave the following ranges:—nitrogen, 2.25-2.35 %; cold water extract, 24-27 %; cold spirit extract, 19.5-23.1 %. The crude fibre in four samples varied 19.2-25.2 %.

MICROSCOPICAL CHARACTERS. Cayenne pepper contains about 1 % of starch similar in appearance to ginger starch. Maize starch has been detected (Hockauf, *P.J.*, 1906, July 28). Wallis has studied the structure of chillies and Japanese chillies, and given drawings (*P.J.*, 1901, Nov. 16; 1902, July 5).

PROSECUTIONS. *Sheffield.* Red lead 6 %. Fine 5s. (*Analyst*, 1881, 6, 153).

Kensington. Dried vegetable tissue devoid of pepper properties 20 %. Fine 10s. (*Grocer*, 1905, April 5; *B.F.J.*, 1905, 100).

GROUND CINNAMON, CASSIA

Cinnamon, known in commerce as Ceylon cinnamon, is at least twice the price of Cassia lignea, known as Chinese cinnamon. There is also an inferior Cassia vera (Stock, *S.P.A.*, 1897, 22, 253).

Hehner (*S.P.A.*, 1879, 4, 225) has pointed out that cinnamon ash contains less than 1 % of Mn_3O_4 , Cassia vera over 1 %, and Cassia lignea up to 5 %, and that Cassia vera yields much less ash than cinnamon. Hendrick (*S.P.A.*, 1907, 32, 14) found that cinnamon contained much more oxalic acid than cassia. The presence of calcium oxalate makes the determination of the ash uncertain, depending somewhat on the amount of ignition; it is better to also carbonate the ash. All the above named, as well as Rau (*Analyst*, 1897, 22, 323) and Winton (*B.F.J.*, 1899, 239), give analyses of cinnamon and cassia.

Of the nineteen samples of *ground cinnamon* examined by Cripps (*S.P.A.*, 1907, 32, 18) fourteen contained less than 1.8 % of ash insoluble in HCl. Bennett (*P.J.*, 1922, May 20) advocated the determination of the total and volatile ether extracts, and gave analyses. He states that there is a loss of volatile ether matter on grinding on a large scale. The microscopic appearance of ground cinnamon is given in the B.P.

ANALYSES OF GROUND CINNAMON, 1928-30

PERCENTAGES.	Usual Range.	Extremes.	Total Samples.
Moisture	8.9-10.5	8.1, 8.2	12
Ash	3.9- 6.0	3.3, 8.2	15
Carbonated ash	4.9- 6.8	4.5, 7.1, 8.2, 8.9, 9.2	17
Ash insoluble in N/2 HCl	0.2- 1.7	2.4 (3), 3.6, 5.8	13
Sand	0.04- 0.6	1.5, 2.4, 3.0, 4.5	13
Cold water extract	5.0- 9.9	10.7	17
Cold methylated spirit extract.	7.6-13.0	14.4, 14.9	17
Ether extract, volatile	0.5- 1.1	0.3, 1.5, 1.7, 2.4	17
.. .. fixed	1.1- 3.3	4.3, 4.5	17
.. .. total	1.7- 4.3	4.8, 5.3	17

Several of the above samples obviously contained an excess of mineral matter.

PROSECUTIONS FOR CINNAMON. *Dumfries.* Ground cassia. It was said by the defence to be "Chinese cinnamon." Fine £1 (*F. & S.*, 1894, Oct. 20).

Aberdeen. Mineral constituents 11.2 %, being 5 % in excess. Fine £3 (*P.J.*, 1903, Nov. 14 ; *B.F.J.*, 1903, 254).

Egremont. Pure cassia. Fine £2 (*Grocer*, 1920, March 5).

Liverpool. Sand and siliceous matter 4 %. The average was said to be 1.0–1.5 %. Fine £5 (*Grocer*, 1920, June 26 ; *B.F.J.*, 1920, 67).

Thornaby-on-Tees. Siliceous matter 3.1 %, instead of only 2.1 % ; the additional matter was practically all sand. The Bench held that the certificate was not adequate, and dismissed the case (*Grocer*, 1924, March 29 ; *Analyst*, 1924, **49**, 228).

Glasgow. Powdered pimento. Fine 12 guineas (*Grocer*, 1921, March 5).

Wood Green. Siliceous matter 5 %, while the average quantity was less than 0.5 %. Fine £2 (*Grocer*, 1925, June 27 ; *B.F.J.*, 1925, 80).

Southport. Sand and siliceous matter 2.5 %. The defendant proved a warranty, and the wholesaler was fined £10 for giving a false warranty (*Grocer*, 1930, June 7 ; *P.J.*, June 7, 21 ; *B.F.J.*, 1930, 68).

Romford. Sand 15 %, being an excess of 13 %. Paid 5 guineas costs (*Grocer*, 1931, Aug. 8).

Bromley. Sand and silicious matter 14.17 %, being an excess of 12.17 %. An explanation was offered that the product was grown in a district of sandy soil, subject to sandstorms. Fine £5 (*Grocer*, 1932, Jan. 23 ; *B.F.J.*, 1932, 15).

PROSECUTIONS FOR CASSIA. *Gateshead.* Spent ginger 30 %. Fine £1 (*F. & S.*, 1895, Jan. 5).

Darlington. Mineral matter 10.3 %, of which 7.3 % was sand. The analyst stated that the sand in fair commercial samples did not exceed 3 %. Fine £1 (*Grocer*, 1905, Dec. 3 ; 1906, 48).

MACE

Mace is obtained from the same tree as nutmeg, and is the coating surrounding the shell of the seed, the kernel of which is nutmeg. E. M. Holmes has given a description with drawings of the structures of the three maces of commerce (*P.J.*, 1908, Nov. 21).

The chief adulterant of the Banda, or cultivated, mace is Bombay, or wild, mace, which is considerably cheaper and has little flavour or aroma. Bombay mace is darker than the genuine article, and is sometimes bleached and then dyed yellow.

Soltsein (*Analyst*, 1897, **22**, 290) has pointed out that the ether extract from Banda mace, which has been previously extracted with petroleum ether, did not exceed 4.8 %, but that Bombay mace yielded about ten times as much. Mitchell and Hackman (*P.J.*, 1909, July 31) have published analyses of thirteen samples of genuine and other mace.

ANALYSES OF MACE

PERCENTAGES.	Samples passed as genuine (15).	Bombay Mace (7).
Moisture	8.6-13.3	4.6- 5.1
Ash	1.6- 3.0	1.3- 2.3
Volatile petroleum ether extract	2.1- 6.4	0.9- 3.5
Fixed petroleum ether extract 11 were	24.0-26.9	27.2-31.6
4 „	27.2-29.6	
Ether extract, after petroleum ether	1.9- 3.7	5 were 30.9-34.3
		1 was 39.7
Cold water extract	7.4-10.5	4.0- 5.5
Cold methylated spirit extract	25.7-33.2	41.0-55.1
B.-R. 40° of fixed petroleum ether extract	70-90	50-89

The greatest contrast is found in the percentages of ether extract after petroleum ether, but the cold water and the cold spirit extracts are also useful. Four samples, considered to be mixtures, are not included in the above table; they had 5.5-7.9 % of ether extract after petroleum ether, and other figures were abnormal.

TEST FOR BOMBAY MACE. Shake about 2 gm. with 20 ml. of methylated spirit for a few minutes; filter. With Banda mace the filter paper when dry is practically colourless. Bombay mace colours the paper yellow, becoming blood-red with N/10 alkali; turmeric, if present, would become brown with alkali (Hanausek, *P.J.*, 1887, Dec.).

PROSECUTIONS. *Burslem.* Ground rice 22 %. The sample was taken in course of transit at the request of the consignee, and the wholesale dealer was fined £40 (*B.F.J.*, 1902, 164).

Wednesbury. Wild mace 30 %. Fine and costs £1 13s. (*P.J.*, 1907, May 4; *B.F.J.*, 1907, 86).

Houghton-le-Spring. Bombay, or wild, mace 26 %. The wholesale dealers stated that the article supplied to the vendor had, unknown to them, been adulterated by the grinders of the spice. Fine £5 (*Grocer*, 1907, Sept. 21; *B.F.J.*, 1907, 156).

London, West. Entirely Bombay mace. To avoid calling expert witnesses to prove the custom of the trade, the summons was withdrawn (*P.J.*, 1909, July 24).

NUTMEG

E. M. Holmes has given an account of the nutmegs of commerce, with woodcuts (*P.J.*, 1909, March 27, April 3).

ANALYSES OF GROUND NUTMEG, 1928-30

PERCENTAGES.	Seven Samples passed Usual Range.	as Genuine. Extremes.	Condemned Sample.
Moisture	—	8.0-10.8	8.2
Ash	2.0- 2.3	2.0- 2.8	2.4
Volatile oil	—	1.5- 3.1	0.8
Fixed oil	—	32.5-38.3	30.0
Cold water extract	8.1- 8.6	7.4-10.7	7.2
Cold methylated spirit extract	18.6-20.6	14.2-24.5	11.3

Two samples of whole nutmeg gave 1.8 % and 1.9 % of ash ; the other figures were similar to ground nutmeg. The largest amount of ash insoluble in acid was 0.08 %.

PROSECUTION. *Cricklade.* Consisted of ground allspice. It was stated that there had been an error in labelling, and the vendor was ordered to pay costs (*B.F.J.*, 1904, 46).

PIMENTO, ALLSPICE

Sometimes, by a misunderstanding, mixed spice for pickling has been supplied as allspice. The L.G.B. Report for 1888 mentions " clove pepper " as a trade synonym for allspice.

The average ash of allspice has been given by Rau (*Analyst*, 1897, **22**, 324) as 6.38 %, by Thamm (*Analyst*, 1906, **31**, 364) as 4.56 % on the dry sand-free substance, and by Winton (*B.F.J.*, 1899, 238) as 4.47 % ; the latter gives the average volatile ether extract as 4.05 %.

PROSECUTIONS. *Wolverhampton.* Ground rice at least 60 %, flavoured with cinnamon and other substances, but no allspice. Fine and costs £2 10s. (*F. & S.*, 1895, Nov. 2).

Wolverhampton. Ground maize 60 %. The defendant supplied " pudding spice," not knowing what allspice was. Fine 5s. (*F. & S.*, 1896, Jan. 4).

Bournemouth. Adulterated with spent allspice. The essential oil was 1.03 %, and the spirit extract 2.65 %, both being below one half of the average figure. The defence attributed the deficiency in essential oil to the article being over four years old. Paid costs (*Grocer* and *P.J.*, 1909, Feb. 6).

CARAWAY FRUIT, OR " SEED "

The B.P. gives a limit of 9 % of ash. Two genuine samples examined by Dyer and Gilbard (*S.P.A.*, 1896, **21**, 207) had 6.1 % and 6.7 %. Another dark-coloured sample contained only 0.1 % of volatile ether extract, as compared with 1.9 % and 1.5 % in the other two ; the fixed ether extract was also low, 16.1 %, as compared with 20 %. The sample had been extracted. Cripps gives 3.07 %

as average of essential oil, and 3.73 % as average of spirit extract (Bournemouth Report, 1907).

PROSECUTIONS FOR GROUND CARAWAY. *London, South-Western.* Extraneous mineral matter 9.3 %. Dismissed owing to warranty (*B.F.J.*, 1924, 118).

Hull. Exhausted of its natural essential and fixed oils to the extent of 65 %. It contained only 6.3 % of essential and fixed oils, instead of at least 18 %. The mineral matter was 15 %, including 4 % of sand instead of not more than 9 % and 1 %, respectively. It was said to have been kept in a drawer for eight years. Fine 2 guineas (*P.J.*, 1927, Sept. 6).

MIXED SPICE. PUDDING SPICE

Although one would expect that mixed spice would contain nothing but spice, rice starch and sugar are both used for making it. As in some spices, such as nutmeg, mace, allspice and cloves, the sandy matter is only about 0.1 %, the amount of sand in mixed spice should be small. The amount of ash in twelve Birmingham samples bought as "mixed spice" or "pudding spice" varied 5.1–6.7 %. The carbonated ashes of eighteen samples varied 3.8–7.9 %, and the sand in twenty-two samples 0.4–1.8 %. These were passed as genuine, but two related samples were condemned as having an excess of sand. Their mean figures were—ash 7.5 %, carbonated ash 8.7 %, and sand 2.4 %.

PROSECUTIONS FOR MIXED SPICE. *Birkenhead.* Spent ginger 20 %. The Government analysts found an excessive amount of sand, but no spent ginger. Costs (*B.F.J.*, 1900, 238).

Liverpool. Spent ginger 15 %. The Government analysts reported 17 %, having found aggregated masses of distorted starch granules. As the defendants denied the possibility of spent ginger being present, and as other analysts failed to find it, the case was dismissed, giving the defendants the benefit of the doubt (*Grocer*, 1905, April 8).

Liverpool. Ground rice 15 %. The grinders declared that the ingredient was a proper one, and the case was dismissed (*Grocer*, 1907, Jan. 5).

Greenwich. Extraneous mineral matter (sand) 2.3 %. As the spice was sold in sealed packets which had not been opened by the defendants, they were held not to be responsible, and the case was dismissed (*Grocer*, 1929, Feb. 2).

DRIED THYME

PROSECUTION. *London, Old Street.* Siliceous matter of the nature of sand 6.5 %, being an excess of 4.5 %. The Public Analyst

said he had found many samples having less than 2 % of sand. Another analyst said many samples contained over 6 % of sand, and that the U.S. limit for medicinal thyme was 4 %. The magistrate considered there was a trivial excess and ordered the defendant to pay 2s. costs (*Grocer*, 1931, July 4 ; *B.F.J.*, 1931, 76).

SAUCES

The writer has given the analysis of a sample of " mushroom ketchup " made from toasted pigs' livers, without any mushrooms (*S.P.A.*, 1904, **29**, 283 ; see also 208).

According to the Preservative Regulations, sauces made from fruit and vegetables may contain benzoic acid up to 250 parts per million, and no other preservative.

Scott Dodd has given analyses of sauces and their minute boric acid content (*S.P.A.*, 1929, **54**, 18).

Of the samples of " sauces " examined in England and Wales during 1906-13, 4.5 % were adulterated, and 1.5 % of the " pickles and sauces " examined 1927-30.

PROSECUTIONS FOR SAUCES. *Belfast.* Salicylic acid 0.025 % Fine 5s. (*Grocer*, 1929, June 8).

London, Guildhall. Crystallised copper sulphate 0.021 %, contrary to the Regulations. The impurity was attributed to the wear and tear of the copper vessels used. As they had been replaced by nickel-lined ones, the summons was withdrawn (*Grocer*, 1929, June 15).

Liverpool. Benzoic acid three times the allowed quantity. Fine £3 (*Grocer*, 1930, March 22).

PROSECUTION FOR CURRY POWDER. *Gillingham.* $\frac{3}{100}$ of 1 % of crushed glass. The glass was attributed to the jagged edge of the cheap glass bottle that contained the powder. The case was dismissed, the magistrates being satisfied it was sold as received. The manufacturers undertook to discontinue the use of such bottles (*Grocer*, 1922, July 1).

CHAPTER XXIX

EFFERVESCENT FOODS AND DRUGS

Baking powder, egg powder; labels, analyses, calculation of composition, baking test, adulteration, analysis, prosecutions. Egg substitute powder. Scidlitz powders, sampling, analysis, prosecutions. Effervescent magnesium sulphate. Effervescent sodium phosphate.

BAKING POWDER. EGG POWDER

BAKING powder is made from sodium bicarbonate and maize, rice or potato flour, with an acid ingredient, which may be— (1) tartaric acid, or cream of tartar, or a mixture of the two; or (2) alum; or (3) an acid phosphate, or, rarely, a mixture of acid substances from two classes. On addition of water carbon dioxide is generated, which takes the place of that produced by yeast fermentation.

Hamill (Food Report, No. 13, to L.G.B. on Baking Powder, 1911) states that rice flour is generally preferred, as maize flour may at times transmit an unpleasant flavour. Maize flour has been found in a number of samples, either alone or mixed with rice. One make of baking powder has a label referring to its "valuable phosphatic contents," while another make is labelled "Guaranteed free from phosphates." Hamill states that a phosphatic baking powder can be produced at about half the cost of a tartaric one. He refers to the generally recognised liability of alum to be injurious to health.

Egg powders are baking powder coloured with turmeric or a coal-tar dye, though a case is on record in which 0.56 % of lead chromate was used (*B.F.J.*, 1917, 75). In 1900 the usual description in Birmingham was "Egg Powder"; in 1916 about half the samples were more correctly labelled "Egg Substitute Powder"; in recent years the word "Substitute" is rarely absent, though sometimes it is in smaller type than "Egg." About half the samples examined were phosphate powders, while about one-third of the samples of baking powder were phosphatic.

The percentages of nitrogen found in fifty-three Birmingham samples were as follows:—0.08–0.49 %, five samples; 0.50–0.89 %, thirty-eight samples; 0.90–1.09 %, ten samples. The proportion of oil varied from 0.2–1.2 %. These figures are similar to those given by rice and maize flours, and show that no egg was present. Eleven Bristol samples examined by Beach, Needs and Russell

(*S.P.A.*, 1921, **46**, 279) showed no evidence of the presence of egg. Two samples of "Real Egg Custard Powders" examined by Hinks (*S.P.A.*, 1923, **48**, 542) contained 0.98 % and 1.39 % of nitrogen, and 6.2 % and 7.1 % of fat, respectively. Each contained dried egg, and the second one dried milk also.

LABELS. Some of the labels attached to egg powders are most misleading. "The use of this preparation will make cakes . . . as light and rich as if fresh eggs were used." "For pastry use half the quantity of butter from your usual recipes." "A perfect substitute for eggs." "Not made from eggs, but is a complete substitute, giving the same lightness, richness and appearance." In some powders there were small coloured masses which looked like dried egg, but were really coloured starch put in to deceive. These powders may give the same "lightness" as eggs, but to claim that they add "richness" or are a "complete" substitute is fraudulent misrepresentation. Sect. 27 of the 1875 Act provides penalties for false labels, and Birmingham manufacturers were cautioned and altered their labels.

ANALYSES. The following table gives analyses of alum and phosphatic powders :—

	PERCENTAGES :	BAKING POWDERS			EGG POWDER	
		A	B	C	D	E
CaO	—	5.1	—	—	6.0
Na ₂ O	8.0	—	25.5	—	4.1
Al ₂ O ₃	3.3	—	—	—	—
Al(Fe)PO ₄	—	—	—	—	1.4
P ₂ O ₅	0.3	2.8	24.3	11.5	6.8
SO ₃	9.2	7.3	—	trace	0.2
Total CO ₂	—	—	17.0	—	4.3
Loss after wetting and drying	15.6	16.2	25.8	12.0	17.0
Ash	20.8	25.5	54.9	26.2	21.7
Ash insoluble in water	—	11.8	—	—	—
Alkalinity of soluble ash as Na ₂ CO ₃	—	2.4	—	—	—

Sample "A," which was marked "Prize Medal," was adulterated with 25 % of alum, and the vendor was fined 10s. for selling an article injurious to health. Sample "B" had an excess of 11.8 % of calcium sulphate and also 25 parts of arsenic per million. The acid calcium phosphate used contained 67 % of CaSO₄. Sample "C" had been prepared from sodium acid phosphate. Sample "D" had a good amount of P₂O₅, but the loss on wetting and drying was small, probably due to decomposition. Sample "E" had less P₂O₅ than "D," but more loss on wetting and drying. The calcium sulphate present amounted to 2 % of the acid calcium phosphate.

Manufacturers have maintained that bread made from baking powder containing calcium sulphate contained sodium sulphate only, all the calcium sulphate being decomposed. Baking experiments by Cripps (*B.F.J.*, 1909, 104) and Cribb (*L.G.B. Food*

Report, No. 13) have shown that this statement is incorrect, as 40–75 % of the CaSO_4 remained undecomposed in the baked loaf (cp. p. 137).

In 1916 the Local Government Board called attention to the presence of arsenic in acid calcium phosphate. Altogether, forty-two Birmingham samples of baking powder and egg substitute powder have been tested for arsenic. Two samples contained 25 parts per million, two had 10 parts, and the remainder not more than 1 part per million.

Acid phosphate of calcium and cream of tartar are much less soluble than tartaric acid, and this may be an advantage by producing slower evolution of carbon dioxide during baking.

The ash of phosphate powders differs from that of tartrate powders in being partly insoluble in water, and giving little or no effervescence with acid. A phosphate powder has a similar alkalinity to methyl orange before and after ignition. The proportion of ash in a phosphate powder is usually higher than in a tartrate one. The following figures show this :—

ASH IN BAKING POWDER

Ash, %	6.2–	10–	15–	20–	25–	30–32.2	Total.
Tartrate powders	35	44	14	1	6	0	100
Phosphate powders	0	0	15	47	34	4	100

Analyses of typical samples of tartrate powders are given below. The moisture has been determined by drying in a vacuum desiccator, as sodium bicarbonate slowly decomposes on drying in the water oven. Gibson (*Analyst*, 1886, **11**, 127) found that the presence of KHT did not affect the rate of loss.

The value of a baking powder depends almost entirely on the amount of carbon dioxide liberated on the addition of cold water. On prolonged drying in the water oven, any NaHCO_3 in excess of that required to neutralise the acid substances present loses half its CO_2 . Experiments by Macara (*S.P.A.*, 1915, **40**, 272) indicate that in certain conditions by boiling with water one-quarter of the CO_2 is evolved, and he considered that this proportion of the undecomposed NaHCO_3 should be added to that liberated by cold water and described as “available” carbonic acid. The author is unaware as to how far other analysts follow this suggestion, and he prefers to avoid the ambiguity of the term “available,” and use the amount of CO_2 actually liberated by cold water by Hepburn’s method, as previously given (p. 75), without correction. Another determination in which excess of acid is used will give the total CO_2 . The difference between the two is generally small. In a number of prosecutions given below the standards used were 8 % of total CO_2 , and 6 % of available CO_2 .

Percentages	BAKING POWDER				EGG SUBSTITUTE POWDER			
	F	G	H	J	K	L	M	N
ANALYTICAL FIGURES :								
Total CO ₂ , % (Q)	14.2	9.6	5.7	5.2	11.6	9.1	6.3	5.7
CO ₂ by water, %	9.2	8.6	4.6	3.7	9.8	6.7	6.3	2.6
Alkalinity, N v/w (L)	93	22	32	64	44	52	*	76
Ash, % (M)	17.8	16.9	14.6	12.3	15.9	11.5	9.7	10.4
Ash alkalinity, N v/w (N)	363	303	269	216	289	218	173	180
Loss on wetting and drying	22.0	20.0	15.9	15.2	22.4	16.8	17.4	13.9
P ₂ O ₅	0.5	—	0.3	0.5	—	—	—	0.2
Baking test :								
Expansion, % 1 to 24	—	61	22	10	—	—	—	10
“ ” 1 to 32	—	—	—	—	85	—	44	—
Price, per oz.	—	—	—	—	1.3 <i>d.</i>	0.8 <i>d.</i>	1.0 <i>d.</i>	1.2 <i>d.</i>
CALCULATED COMPOSITION :								
Moisture	6	7	8	9	7	8	9	9
Sodium bicarbonate	25	18	11	10	22	17	12	11
Magnesium carbonate	3	—	—	—	—	—	—	—
Tartaric acid	20	11	6	0	14	12	9	0
Cream of tartar	0	10	4	10	5	0	6	10
Sodium tartrate	0	4	14	5	0	2	0	0
Dry flour (by difference)	46	50	57	66	52	61	64	70
	100	100	100	100	100	100	100	100

* Acidity, N v/w 11.

Calculation of Composition. When slightly damp materials are used in preparing the powders, or when they are exposed to damp air, decomposition will take place, CO₂ being liberated. In these conditions Na₂CO₃ cannot be present, and owing to the greater solubility of H₂T̄ than KNaT̄, formation of Na₂T̄ is much more likely than that of KNaT̄, and there has been no indication of the presence of the latter in somewhat decomposed powders. On these assumptions the following equations have been used to calculate the approximate composition of the powders given above. As the results depend on differences, logarithms of the factors have been given, so that analytical errors shall not be magnified by arithmetical inexactitude.

The percentage of total CO₂ is indicated by “Q” and that of ash by “M,” the Nv/w of the powder by “L,” and that of the ash by “N,” as previously stated. Of course, the logarithms of these values must be added to the logarithms of the factors given. “T” “C” and “S” are used for H₂T̄, KHT̄ and Na₂T̄, 2 Aq., respectively. The percentage of NaHCO₃ = 1.909 Q. The equations will be more or less inapplicable, in the presence of other substances than those named. Sodium bicarbonate is assumed to be always present.

- (i.) H₂T̄ % (with C and S) = 1.3936 N + 0.2318 Q —
2.8752 L — 0.6683 M
- (ii.) H₂T̄ % (with S only) = 0.02318 Q — 2.7852 L
- (iii.) H₂T̄ % (alone) = 2.8752 (N — L)

- (iv.) $\text{KHT} \bar{\%}$ (with T and S) $= 1.0675 \text{ M} - 1.7918 \text{ N}$
 (v.) $\text{KHT} \bar{\%}$ (with S only) $= 0.6310 \text{ Q} - 1.2745 \text{ L}$
 (vi.) $\text{KHT} \bar{\%}$ (with T only) $= 1.2745 \text{ N} - 0.6310 \text{ Q}$
 (vii.) $\text{KHT} \bar{\%}$ (with T only) $= 0.4350 \text{ M} - 0.5158 \text{ Q}$
 (viii.) $\text{Na}_2\bar{\text{T}}, 2 \text{ Aq. } \bar{\%}$ (with T and C) $= 1.6933 \text{ N} - 0.4174 \text{ Q} - 0.8539 \text{ M}$
 (ix.) $\text{Na}_2\bar{\text{T}}, 2 \text{ Aq. } \bar{\%}$ (with T only) $= 1.0608 \text{ N} - 0.4174 \text{ Q}$
 (x.) $\text{Na}_2\bar{\text{T}}, 2 \text{ Aq. } \bar{\%}$ (with T only) $= 0.3365 \text{ M} - 0.4174 \text{ Q}$

The equations (i.), (iv.) and (viii.), which are applicable when the three substances are all present, should be first used, and if one or other of the constituents is indicated to be absent, other equations can be used. If serious minus quantities appear, or two equations do not approximately agree, an analytical error is present, or a fourth substance. Baking powder "F," above, gave -17% $\text{KHT} \bar{\%}$, and further examination detected about 3% of magnesium carbonate.

Having calculated the approximate composition, it is a useful check to calculate back to the analytical figures by the following equations: "B" indicates percentage of NaHCO_3 :—

- (xi.) $\text{L} = 11.9 \text{ B} - 13.3 \text{ T} - 5.3 \text{ C}$
 (xii.) $\text{M} = 0.63 \text{ B} + 0.37 \text{ C} + 0.46 \text{ S}$
 (xiii.) $\text{N} = 11.9 \text{ B} + 5.3 \text{ C} + 8.7 \text{ S}$

Baking powder "F" was a good tartaric acid powder containing magnesium carbonate, with no decomposition. "G" had tartaric acid and cream of tartar and a little sodium tartrate. "H" was of poor quality; it was caked, and the high sodium tartrate (14%) also pointed to decomposition. "J" was an inferior powder containing two-thirds of dry flour, and was caked; the original tartaric acid had become sodium tartrate. The vendors of these two samples were cautioned.

Egg substitute powder "K" was of good quality. "L" was of medium quality, prepared by the manufacturers of baking powder "J." "M" was of medium quality and differed from the others in having an excess of acid present. "N" was of very poor quality, containing 70% of dry flour. The absence of sodium tartrate showed that the inferiority was due to its ingredients, and not to decomposition.

There is little variation in the proportions of moisture in the samples, and the figures had little correlation with the CO_2 liberated by water.

A rough indication of the quality of a baking or egg powder may readily be obtained by wetting it, evaporating the water, and drying till constant in weight. The amount of loss should be at least 16% ; 62% of the samples lost at least that amount. Correction by subtraction of the amount of moisture (determined in the vacuum

desiccator) will give approximately the carbonic acid (H_2CO_3) lost, which should not be less than 10 %. About two-thirds of it is CO_2 liberated by water.

Baking Test. Egg substitute powder "N" was of such inferior quality, yielding only 2.6 % of CO_2 by water, that the vendor was prosecuted, and baking tests were made with it and other samples. According to the directions, one heaped-up teaspoonful was to be added to $\frac{1}{2}$ lb. of flour. It weighed about $\frac{1}{2}$ oz., or a proportion of 1 to 32 of flour. These quantities of powder and flour were rapidly mixed with 5 oz. water, transferred to a tin and baked for about an hour at about 200°C . At the same time flour without powder was baked in the same conditions to ascertain the expansion produced by the powder. In one experiment flour alone gave a loaf having a volume of 390 ml., and egg substitute powder "M" 560 ml., being an expansion of 44 %. The powder in question, "N," when rather more (1 to 24) was used only gave an expansion of 9 % and 14 % in two trials. The manufacturer admitted that he made a better powder. The price charged for "N" was, however, more than that charged for the better powders "M" and "L" of other makers.

Although there had been no previous prosecutions for egg substitute powder of deficient strength, it seemed reasonable to expect the strength to be equal to the similar substance, baking powder. The magistrates dismissed the case on the grounds that scientific evidence alone was not sufficient to enable them to fix a standard. The reason given was unsatisfactory, as both users and vendors would be ignorant as to the amount of carbon dioxide that should be present, and manufacturers might not know the actual yield.

ADULTERATION. Of the seventy-six samples of baking powder examined in Birmingham from 1900 to 1928, six contained alum, three excess of calcium sulphate, and four were deficient in carbon dioxide.

None of the seventy-eight samples of egg, or egg substitute, powder were adulterated with alum; but five contained calcium sulphate in excess, three were deficient in carbon dioxide, and fifteen had false labels.

According to the official reports for England and Wales, no less than 27 % of the samples of baking powder examined in 1893 were adulterated, generally with alum. During 1902-13, the proportion had fallen to 4.6 %, and during 1919-30, to 2.3 %, while 1.4 % of the samples of baking mixtures, 1920-30, were condemned. Of egg powders and substitutes 3.4 % were reported adulterated, 1919-30.

ANALYSIS. The *alkalinity* of the powder is determined by boiling 0.5 gm. in a flask with 100 ml. water and 25 ml. or more N/10 HCl for fifteen minutes. Titrate back, using phenol phthalein for tartrate powders and methyl orange for phosphate powders.

The use of a larger quantity than 0.5 gm. makes the reading of the end point more difficult. With egg powders comparison of the colour with 0.5 gm. in water is useful. Petroleum ether should be used for extracting the *fat*. If ether is used, two-thirds of the extract may be tartaric acid. *Sulphates*, due to alum or calcium sulphate, if detected, should be determined on the powder, and not on the ash. Methods for the determination of carbonate and phosphate have been previously given (pp. 75, 73). The accidental substitution of *oxalic acid* is possible and would be serious. Add water, filter, neutralise and add CaCl_2 solution. Let stand twenty minutes, and boil precipitate with weak $\text{H}\bar{\text{A}}$. Oxalic acid gives an immediate fine precipitate, insoluble in $\text{H}\bar{\text{A}}$. Tartaric acid forms large spherules slowly. They are insoluble in $\text{H}\bar{\text{A}}$, and are charred on ignition.

Details of American methods of analysis have been given by Crampton (*Analyst*, 1890, **15**, 26).

PROSECUTIONS FOR BAKING POWDER. *Cambridge.* A baker having been fined for selling buns containing alum the vendor of the baking powder used, about one-third of which was alum, was prosecuted for selling an article injurious to health. The vendor was fined £2 and appealed to Quarter Sessions. The Recorder quashed the conviction, as he thought baking powder was not an article of food, and that bread made with it was not injurious to health. He allowed £100 costs, to be paid by the Authority (*Analyst*, 1879, **4**, 176, 231; 1880, **5**, 21).

Tynnewydd. Alum 54 %, in an American preparation. Fine 33s. (*F. & S.*, 1893, Jan. 28).

Pontypridd. Alum 39 %. The vendor was fined £2 for the sale of an article injurious to health, and appealed to Quarter Sessions, when the conviction was confirmed, after five days' hearing. On appeal to the High Court, *James v. Jones* (1894), the conviction was quashed, it being held that baking powder was not an article of food (*Analyst*, 1893, **18**, 152; 1894, **19**, 48; *F. & S.*, 1893, Feb. 25, April 15, Dec. 23).

Llantrissant. Alum 60 %, sodium bicarbonate 26 %, rice starch 14 %. The quantity of sodium bicarbonate was insufficient to neutralise the alum. Fine £1 (*F. & S.*, 1893, Sept. 16).

Birmingham. Alum 25 %. Fine 10s. for sale of an article injurious to health (*B.F.J.*, 1900, 337, 371).

Norwich. Crystallised alum 37 %. It was labelled "This packet is warranted to contain sulphate of aluminium and potassium." A 4-lb. loaf made with the baking powder, according to the directions, would contain 160 grains of alum. The Medical Officer of Health considered that the prevalence of constipation in Norfolk was largely due to such bread. Fine 5s. only, as the Bench thought the vendor had been misled (*B.F.J.*, 1901, 210).

Norwich. Crystallised alum 42.7 %. The case was dismissed,

as the summons, instead of stating "alum, which rendered the baking powder unfit for food," said "alum, which was injurious to health" (*B.F.J.*, 1903, 141).

Hull. Alum 45.8 %, 50.2 %, 50.0 %, respectively. The summonses were withdrawn, as the addition was acid phosphate of lime.

Lowestoft. Total carbonic acid 4.1 %, available carbonic acid 0.85 %, instead of 8 % of total carbonic acid gas, of which 90 % should be available. It was practically devoid of available carbonic acid, the essential ingredient. Fine £2 (*Grocer*, 1907, July 27, Aug. 3; *B.F.J.*, 1907, 138).

Lambeth. Available carbon dioxide 2.4 %, instead of at least 6 %. For the defence a smaller packet of another make, sold at the same price, was shown, and the Public Analyst admitted that the two packets had the same amount of rising matter. Case dismissed and the defendant allowed 2 guineas costs (*Grocer*, 1914, Sept. 12; *B.F.J.*, 1914, 168).

Bolton. Plaster of Paris 33.6 %, arsenic 1.75 grains per lb., instead of less than 6 %, and $\frac{1}{100}$ grain, respectively. Fine £2 (*Grocer*, 1916, Dec. 2; *B.F.J.*, 1916, 462).

Newcastle-on-Tyne. Ground rice 80 %, calcium superphosphate 4 %, sodium bicarbonate 6 %, calcium and sodium phosphates and moisture 10 %. The ingredients were only sufficient to yield 2.6 % of carbon dioxide, instead of 6 % as a minimum. The Public Analyst considered the ground rice to be in excess, but admitted it was similar to typical recipes quoted by Hamill (*Food Report*, No. 13, p. 8). Case dismissed (*Grocer*, 1916, Oct. 14; *B.F.J.*, 1917, 16).

Mansfield. Carbonic acid gas 4.33 %, instead of 8 %. A chemist for the defence said that the deficiency in gas did not prove that the powder was deficient in quality, as two cakes had been baked with it and the results were satisfactory. Several women witnesses stated that they had used the powder for several years and were satisfied with it. Dismissed (*Grocer*, 1917, Dec. 8).

Havant. Salt 5.27 %, which injured the gas-producing qualities of the other ingredients. Fine 5s. (*Grocer*, 1919, March 22).

West Ham. Available carbon dioxide 1.1 %, and of very little value as a baking powder. Fine and costs 61s. (*Grocer*, 1923, June 16).

West Ham. Available carbon dioxide 0.4 %; it was useless as a baking powder. For the defence it was argued that neither the summons nor the certificate was sufficient, as they did not say why it was useless, but the objection was overruled, and the sample was sent to the Government Chemist. His report was: "The sample was enclosed in a paper wrapper, a condition of packing which might result in the loss of carbon dioxide during the time that had

elapsed since the sample was taken." Prosecution withdrawn (*Grocer*, 1926, Feb. 5).

Hedingham. A grocer was fined £5 for selling a baking powder which contained 9 parts of arsenic per million. His wholesaler paid his fine, and took action against the chemical merchant who had supplied him with acid calcium phosphate containing about 20 parts of arsenic per million. He stated that the baking powder was made from equal parts of acid calcium phosphate, bicarbonate of soda, and rice flour. The Judge found that 4 parts of arsenic per million in the phosphate was the outside limit, and that the excess constituted a breach of warranty. He awarded damages of over £71 (*Grocer*, 1928, June 21, 30).

Wolverhampton. Carbon dioxide 1 %, whereas a reasonable percentage was 8 %. For the defence it was stated that the article was sold in unopened packets, but that it had been in stock about four years. Paid costs 53s. 6d. (*Grocer*, 1929, June 8).

Saffron Walden. Giving a false warranty with baking powder which contained $\frac{1}{35}$ grain of arsenic per lb. The defendant stated that he believed his warranty to be true, as he obtained warranties with his ingredients, but had not had any analyses made. He was given the benefit of the doubt and the case was dismissed (*Grocer*, 1930, Jan. 18, Feb. 15; *B.F.J.*, 1930, 14).

PROSECUTIONS FOR EGG POWDER. *Godstone*. Alum 28 %. Fine £1 (*B.F.J.*, 1900, 205).

Swindon. Arsenic $\frac{1}{40}$ grain per lb. Fine £10 (*B.F.J.*, 1918, 83).

Birkenhead. Sulphate of lime 41.3 %. It bore a label stating that it was of the highest quality, and could not be equalled in value. The Public Analyst stated that it was purely a baking powder, with nothing of the nature of eggs in it. Fine £3 (*Grocer*, 1918, Nov. 16).

London, Tower Bridge. No trace of egg. It should have been labelled "Egg substitute." It contained maize flour 63 %, sodium bicarbonate 20.6 %, tartaric acid and a trace of colouring matter 16.4 %. Fine and costs 41s. (*Grocer*, 1926, April 17; *Analyst*, 1926, 51, 299).

Saltash. Tartaric acid, sodium pyrophosphate, bicarbonate of soda, rice starch and a yellow dye, but no egg. It was labelled "Real egg powder, double strength, guaranteed to contain the real yolks of eggs." The Bench considered the retailer had no intention of committing an offence, and fined him 10s. only (*Analyst*, 1931, 56, 661; *P.J.*, 1931, Sept. 5).

PROSECUTIONS FOR EGG SUBSTITUTE POWDER.

Hartlepool. Calcium sulphate 4.26 % in excess, the phosphate powder used containing 35 % of calcium sulphate, instead of 10 %. The Medical Officer of Health considered the daily use of the powder would lead to cumulative ill-effects. For the defence, it was claimed

that there was no legal definition of "substitute," and that the public was satisfied with it. A public analyst compared the article with a proprietary brand of ale containing 12·4 grains of calcium sulphate per pint. Fine £10 (*Grocer*, 1919, Nov. 22; *B.F.J.*, 1920, 6).

Birmingham. Carbonic acid gas liberated by water 2·5 %, instead of at least 6 %. The manufacturer stated that he made two qualities, and that he had had no complaints made about this, the cheaper one. The magistrates dismissed the case on the grounds that scientific evidence alone was not sufficient to enable them to fix a standard (*Analyst*, 1927, **52**, 536) (*cf.* p. 438).

Witham. Arsenic $\frac{1}{8}$ grain per lb. Fine £10 (*B.F.J.*, 1929, 19).

SEIDLITZ POWDERS

In 1815 T. F. Savory obtained a patent, which was subsequently declared invalid, for "Seidlitz powders" which he claimed had all the properties of the spring at Seidlitz, Germany. Each dose was stated to be tartaric acid 40 grains dissolved in $\frac{1}{2}$ pint of spring water, with the addition of Rochelle salt 120 grains, and sodium bicarbonate 40 grains, previously mixed.

They were introduced into the B.P. in 1890, and in 1914 the weights were given in grammes, in each case with a slight alteration in quantity. "Extra strong" and "Double strong" powders are not mentioned by the B.P., but are contained in the B.P. Codex.

COMPOSITION OF SEIDLITZ POWDERS, BEFORE AND AFTER MIXING

Grammes.	B.P.	Extra Strong.	Double Strong.
WHITE PAPERS :			
Tartaric acid	2·5	2·5	2·5
BLUE PAPERS :			
Sodium bicarbonate	2·5	2·5	2·5
Rochelle salt (KNaT, 4H ₂ O)	7·5	11·25	15·0
Total weight	10·0	13·75	17·5
Sodium bicarbonate %	25·0	18·2	14·3
COMPOSITION AFTER MIXING :			
Sodium tartrate (Na ₂ T, 2H ₂ O)	3·42	3·42	3·42
Total tartrate	10·92	14·67	18·42
Tartaric acid in excess	0·27	0·27	0·27

When a small quantity of water is used an opalescence may be present, due to the formation of cream of tartar from the excess of tartaric acid.

During the years 1919–29 there were 148 purchases in Birmingham of "Seidlitz powders," in which the alkaline powders weighed under 12 gm.; in most cases two powders were bought; particulars are given below :—

ANALYSES OF SEIDLITZ POWDERS

WHITE PAPERS :							
Grammes	. 0.4-1.9	2.0-	2.2-	2.4-	2.6-	2.8-3.6	Total.
Percentage of samples	. 2	3	19	63	9	4	100
BLUE PAPERS :							
Grammes	. 8.8-9.2	9.4-	9.8-	10.2-	10.6-	11.0-11.4	Total.
Percentage of samples	. 1	6	71	17	4	1	100
Percentage of NaHCO_3	. 16.2-19.0	23.6-	24.0-	26.0-	28.0-	28.5	Total.
Percentage of samples	. 2	3	89	4	2		100

The above figures show that, apart from carelessness or accident, there is no practical difficulty in supplying powders in which the weight of the alkaline powder shall be within 5 %, above or below, of the standard 10 gm., and the acid powders within 12 %. The alkaline powders also should not be outside the limits of 24-26 % of sodium bicarbonate.

In the same years, 1919-29, thirteen samples were either sold as "Extra strong" or having alkaline powders weighing over 12 gm. were probably intended to be such. The composition of these is given below :—

ANALYSES OF "EXTRA STRONG" SEIDLITZ POWDERS

WHITE PAPERS :					
Grammes	. .	1.4-2.3	2.4-2.6	2.9-3.2	Total.
Percentage of samples	. 16	60	24		100
BLUE PAPERS :					
Grammes	. .	12.1-12.5	13.0-	14.0-14.5	17.9-18.5
Percentage of samples	. 27	42	23	8	100
Percentage of NaHCO_3	. 8.4-12.0	17.0-	18-20	24-25	
Percentage of samples	. 15	39	31	15	100

The above results are much less satisfactory than those found for the B.P. powders, and indicate that, while powders complying with the B.P. Codex standard preponderate, other vendors ignore or are ignorant of it. The presence of 25 % of sodium bicarbonate in some of them is probably due to the vendor using the trade preparation "Pulv. Seidlitz" of that composition, under the misapprehension that the use of 13.75 gm. of that mixture would give the correct article. Such a powder, unless the acid be proportionately increased, would be objectionably alkaline, instead of pleasantly acid.

One interesting sample of "Extra strong" powders had the following composition :—Tartaric acid 3.0 gm., instead of 2.5 ; sodium bicarbonate 3.2 gm., instead of 2.5 ; Rochelle salt 9.6 gm., instead of 11.25. Although each quantity was incorrect, the mixed draught would differ little from that of standard composition, owing to the extra sodium tartrate formed being almost equivalent to the

deficiency of Rochelle salt. Saccharin was detected in a sample which was labelled "sweetened."

There appears to be little demand for "Double strong" powders; the single sample examined was in close agreement with the B.P. Codex standard.

During the period 1890-1918, of the 182 Birmingham samples examined, no less than 31 % were condemned. In many cases the defects were probably due to carelessness, but in others fraudulent substitution of cheaper ingredients for Rochelle salt had been practised, or there was a marked deficiency in the weight of tartaric acid. The most common substitution was sodium bicarbonate, resulting in a strongly alkaline powder being produced; in one instance sugar was added to make it look more bulky. In one case 20 % of anhydrous Glauber salt, and in another 26 % of Epsom salt, had been substituted for Rochelle salt. The worst cases were due to unqualified hawkers who supplied huckster's shops. A number of vendors were cautioned or prosecuted, and in the following period, 1919-29, the percentage of adulteration fell to 10, and the samples were incorrect from carelessness rather than fraud.

Of the samples examined in England and Wales during 1898-1903, 14.6 % were condemned; during 1904-13 the percentage had fallen to 7.2, and during 1920-30 it had again risen, being 10.0 %.

SAMPLING. The question of the proper method of division of a purchase of Seidlitz powders has given rise to a considerable amount of discussion. In 1900 the Editor of the *British and Colonial Druggist* maintained that weighing was not analysis, and therefore no division of the sample was necessary. It is obvious that if an inspector empties powders from their papers and mixes them, any checking of the weight by the Public Analyst is impossible, and fraudulent vendors, if the composition is correct, may with impunity sell any weight they like.

In the writer's opinion, which has been upheld by the Birmingham magistrates, a box of Seidlitz powders should be bought, and the contents dealt out like cards in three packs, each of which forms a sample. The method was tested by weighing each of the twelve powders in a box. The alkaline powders varied in weight from 9.7-10.3 gm., and the true average was 10.08 gm. The average weight of the first, fourth, seventh, and tenth powders was 10.13 gm., of the second pack 10.08 gm., and of the third pack 10.02 gm. Similarly, the acid powders varied 2.13-2.76 gm., and their true average was 2.42 gm. The average of the first pack was 2.45 gm., of the second 2.45 gm., and of the third 2.36 gm. It will be seen that the extreme differences in the packs are about 0.1 gm., a quantity much too small to be of practical importance.

This method will differ from that found incorrect in the appeal case *Mason v. Cowdary* (see p. 21), in that the powders being taken

from one box will all be from one maker ; also, it is the only possible one of enforcing the Act. Informal samples should be of two or three powders.

ANALYSIS. *White papers.* Weigh 1.2 gm., add 30 ml. N/2 NaHO (standardised to phenol phthalein) from a pipette, and complete the titration. $\% \text{H}_2\text{T} = \text{ml. used} \times 3.126$.

Blue papers. Weigh 3.0 gm., add 20 ml. N/2 HCl and thoroughly boil off CO_2 . Add phenol phthalein and titrate back with N/2 NaHO. $\% \text{NaHCO}_3 = \text{ml. used} \times 1.4$. Weigh 2 gm., ignite till fumes cease to escape. Add 35 ml. N/2 HCl, filter and wash into flask, boil and titrate, using methyl red. Subtract from the number of ml. used two-thirds of the former titration, and $\% \text{Rochelle salt} = \text{difference} \times 3.528$.

Neither powder should contain more than a trace of sulphate ; sugar may be tested for by Fehling solution after inversion.

PROSECUTIONS. *Glasgow.* Tartaric acid deficiency of 15 grains, sodium bicarbonate 170 grains in excess, sodium sulphate 21 grains, sugar 56 grains, while Rochelle salt was absent. Fine 30s. (*P.J.*, 1891, June 20).

Mansfield. Blue papers contained 133 and the white papers 37 grains. It was suggested by the defence that the discrepancies were due to the atmosphere. Costs 25s. (*F. & S.*, 1895, March 9).

West Bromwich. Average deficiency of the blue papers 9.1 grains, and excess of $3\frac{1}{2}$ grains in the white papers. The Public Analyst stated that he mixed all the powders together and then made the analysis. The stipendiary thought each packet should have been divided before being analysed, and dismissed the case (*F. & S.*, 1896, Feb. 29).

Castle Eden. Deficiency of 30 % of tartaric acid. The other packet was genuine. Fine 1s. (*F. & S.*, 1899, Jan. 28).

Loughborough. Alum 12 % in the acid powders. Fine £1 (*B.F.J.*, 1899, 280).

Birmingham. Tartaric acid only 47 %, sodium potassium tartrate only 33 %, and sodium bicarbonate 213 %, of the quantities required by the B.P. The box was labelled "CAUTION TO THE PUBLIC. Thousands of boxes of a common imitation of the genuine Seidlitz Powders are being sold by unprincipled traders for the sake of extra profit. We guarantee all our powders to be genuine." Fine £10 (*B.F.J.*, 1900, 85).

Birmingham. The contents of the blue papers varied from 118–153 grains, 81 % being sodium bicarbonate and the remainder sugar. The white papers contained 20–41 grains of tartaric acid. The powders were labelled "Although each ingredient is HANDWEIGHED, the weight of the contents cannot be guaranteed to a few grains. ACCIDENTS may occur in WEIGHING, or loss keeping in stock, however careful." The powders were bought from

a huckster's shop. A sample from another huckster's shop with the same label had blue papers which contained 107-133 grains of sodium bicarbonate only. Subsequently a sample was obtained by the inspector from the unqualified hawker who supplied these shops, and a shopkeeper submitted a sample obtained from him for analysis. These two were of similar composition to the second sample. The cases were not heard, as the hawker absconded (1910 Report).

Birmingham. Sodium bicarbonate 83 %, dried Epsom salt 17 % in the blue papers. It was labelled "Seltzer powder" in small letters and "SEIDLITZ POWDER" in capital letters. Fine £2 (1912 Report).

Portsmouth. Acetanilide 50 %, sodium bicarbonate 47.6 %, moisture, etc., 2.4 %. The defendant pleaded that a girl employed by him had used an ingredient of a headache powder by mistake, and said that the mistake had then, in compensation paid, and withdrawal from sale, cost him £100. Fine £5 (*P.J.*, 1914, Sept. 12; *B.F.J.*, 1914, 179).

EFFERVESCENT MAGNESIUM SULPHATE

This article is required to contain 50 % of magnesium sulphate. The "Additions" of 1890 first introduced it to the B.P. A similar article containing no citric acid has been popularly, but incorrectly, described as "citrate of magnesia"; "effervescent magnesia" is a better description. A Birmingham sample examined in 1915 yielded 17.8 % of ash. According to Davidson and Lunan (*P.J.*, 1899, Feb. 18), about one quarter of the carbon dioxide in the sodium bicarbonate is lost during preparation.

PROSECUTION FOR CITRATE OF MAGNESIA. *Greenock.* Composed of sodium carbonate and sulphate, tartaric acid, and sugar. Evidence for the defence was given that the article was commercially known as "citrate of magnesia," and a medical witness stated it was the article he intended when he ordered "citrate of magnesia." Case dismissed (*C. & D.*, 1875, Nov. 15).

EFFERVESCENT SODIUM PHOSPHATE

According to the B.P., this article contains the equivalent of 50 % of sodium phosphate, made effervescent by the addition of sodium bicarbonate, tartaric and citric acids.

PROSECUTION. *London, Marylebone.* Two samples were certified to contain $8\frac{3}{4}$ grains, and $3\frac{1}{2}$ grains, respectively, of white arsenic per lb. The Medical Officer of Health pointed out that $\frac{1}{2}$ oz., a maximum dose, would contain $\frac{1}{4}$ grain of arsenic, and be within dangerous doses. The Public Analyst considered the impurity was in the form of sodium arsenate. For the defence, it was pleaded

that the article conformed with the Pharmacopœia requirements, as no test for arsenic was given there ; the source of the impurity was considered to be the commercial sulphuric acid used for making the sodium phosphate. The magistrate stated that, although the presence of arsenic was never suspected, the vendors were responsible, and he fined them £10 each, and costs (*C. & D.*, 1900, July 14 ; *B.F.J.*, 1900, 237).

CHAPTER XXX

MISCELLANEOUS FOODS

Infants' foods. Lemon cheese and curd. Ice cream. Yeast. Table salt. Other foods.

INFANTS' FOODS

Food Report No. 20 of the L.G.B., 1914, by Coutts and Baker, contains a full discussion of the subject, both from a medical and analytical point of view. It gives many examples of proprietary foods, with analyses, and methods of analysis. More recently analyses of various foods have been given by the Queensland Government Analyst (*Analyst*, 1925, **50**, 21).

A few examples of false labels on samples bought in Birmingham may be given :—"The most perfect substitute for mother's milk. . . . It contains all the nutritive elements in approximately the same proportion in which they exist in this secretion." The relation in human milk of the saline, albuminous and carbohydrate constituents is about 1, 9, and 30 ; in this food the relationship was about 1, 16, and 110, and the constituents were not the same.

"Rich in those phosphatic and nitrogenous constituents so necessary for the formation of flesh and bone." The phosphate in wheat flour is about 0.4 % ; this food had 0.5 %. Wheat flour has about 1.9 % of nitrogen ; this food had 1.7 %. The food therefore had only the richness of ordinary wheat flour.

"Avoid foods composed of starch only." The food contained over 80 % of starch.

PROSECUTIONS. *Liverpool.* Wheat starch. The tin was labelled "—— Food only should be used in infant life, being free from adulteration, such as starch, gluten, malt, etc." The two points made by the prosecution were that the food was sold as being free from starch, and that it contained such a proportion of starch as to be useless and even injurious to the infant. Twenty-six infants, said to have been delicate, were in court to show the beneficial effects it had had on them. The defendant was stated to have sold very many tins of the food and never to have had a complaint. The stipendiary was not satisfied that the food was injurious, or unfit for infants. It had also been proved that the starch present was natural to the food and had not been added as an adulterant. He dismissed the case with costs (*P.J.*, 1911, April 1).

Rutland. Practically unaltered starch upwards of 70 %. The Medical Officer of Health considered the food would be deleterious

to the health of young children, and that following the directions given would not cause any extensive change from starch to sugar. A mother deposed that she had brought up two of her children on the food with the best results. The defendant stated that he had sold the food for twenty-three years without complaint. Fine 10s. (*B.F.J.*, 1912, 95).

LEMON CHEESE. LEMON CURD

These articles were originally prepared from eggs, butter, sugar and lemon, and the Queensland Regulations require these ingredients only to be present in lemon cheese (*Analyst*, 1927, **52**, 31, where analyses are given). Very inferior products containing margarine, glucose, British gum, tragacanth gum, and an excessive amount of water have been sold as lemon cheese. Papers by Elsdon (*S.P.A.*, 1925, **50**, 230) and Hodgson (*S.P.A.*, 1925, **50**, 396) should be consulted.

Of the samples of these articles bought in England and Wales, 1920-30, 11·8 % were reported to be adulterated.

PROSECUTIONS FOR LEMON CHEESE. *Birkenhead*. Starch and added water 42 %, being excessive amounts. Case dismissed as analyst's certificate was bad in law (*Grocer*, 1925, Dec. 19 ; *Analyst* 1926, **51**, 84, 243 ; *B.F.J.*, 1926, 4).

Kensington. Salicylic acid 0·63 grain per lb., contrary to the Preservative Regulations. The vendor undertook to withdraw the article from sale, and the summons was withdrawn (*B.F.J.*, 1927, 105).

PROSECUTION FOR HOME-MADE LEMON CHEESE. *Salford*. Deficient in butter, sugar, and eggs. There was no butter, not more than a trace of eggs, 26 % of cane and invert sugar, with 39 % of glucose syrup, artificial colouring, starch, and 8 % of water in excess of the normal. The retailer was fined £5 for its sale, and the makers £5 and £1·5 special costs, for the use of the false label " Home-made," which the stipendiary considered meant made of articles which would be used in a domestic household (*Analyst*, 1929, **54**, 105 ; *B.F.J.*, 1929, 7).

PROSECUTION FOR LEMON CURD. *East Dereham*. Salicylic acid 1·5 grains per lb. Evidence was given for the defence that there was no standard, and that a preservative was necessary. Case dismissed (*Grocer*, 1907, Sept. 21 ; *B.F.J.*, 1907, 174).

Barton-on-Humber. Boric acid 0·5 %. The case was dismissed as the analyst's certificate did not give the whole component parts, but only the parts of foreign matter (*Grocer*, 1913, Oct. 18).

Hull. Starchy material prepared with water, with small quantities of butter and lemon flavouring, and coloured with aniline dye. Fine £2 (*P.J.*, 1913, Dec. 20).

ICE CREAM

Prosecutions are recorded below for the presence in ice cream of zinc, boric acid, and excessive numbers of bacteria, but the writer is only aware of prosecutions under local Acts to set up a standard of composition.

The Report of the Ministry of Health for 1924 quoted the Poplar Public Analyst as stating, "The designation 'ice cream' appears to be very loosely and irregularly applied to a variety of products which may vary from sweetened water to sweetened cream," and being of opinion that a vendor selling as "ice cream" something devoid of cream should be dealt with as committing an offence.

G. D. Elsdon found that nine samples of "cream ice" contained 1.6.7 % of butter-fat, and two others 9 % and 18 %, respectively. He suggested adopting a United States standard for "ice cream" of 12 %. The vendors of the unsatisfactory samples were cautioned (1925 Salford Report).

Of twenty-one samples of "cream ice" bought in Portsmouth, examined by Page, ten had 10 % of fat and over, six had 5 % and over, and five had under 5 % of fat (1926 Report).

In 1928 Tankard examined twenty samples of "ice cream" chemically. The amount of milk fat found was 1.0-4.4 %, instead of 6-8 %; they were not properly named, as there should have been some addition of cream. The bacteriological examination of twenty-nine samples showed that thirteen of them were contaminated with objectionable organisms (*Analyst*, 1929, **54**, 661).

The limitations of the Babcock method for determining fat have been discussed by Bahlman (*Analyst*, 1915, **40**, 442) and Utt (*Analyst*, 1915, **40**, 461), and Remington and McRoberts have described a method for determining gelatin (*Analyst*, 1927, **52**, 288).

During 1897-9, 5.9 % of the samples examined in England and Wales were reported adulterated; during 1903-13 the proportion fell to 1.7 %, and rose to 4.0 % during 1920-30.

PROSECUTIONS FOR ICE CREAM. *Brentford.* Zinc 0.01 %. Vendor ordered to pay costs (*F. & S.*, 1896, Sept. 19).

Southwark. A million microbes per ml., and injurious to health. They were attributed to the use of decomposing milk. Fine £10 (*F. & S.*, 1898, Oct. 22).

Southwark. Micro-organisms 28 millions per ml., including at least 200 *Bacillus coli communis*, and injurious to health. The case was dismissed as the Public Analyst had not personally made the determination (*B.F.J.*, 1899, 307).

Cromer. Boric acid 8.7 grains per lb. The Medical Officer of Health described it as a useless and silly addition to a frozen article. The magistrates were not satisfied that the preservative would be

injurious to health, and dismissed the case (*Grocer*, 1923, Sept. 22; *B.F.J.*, 1923, 87).

Bangor (Ireland). Milk fat 3.2 %, instead of at least 5 % as required by the Bangor Borough Council Act. At least $\frac{3}{4}$ pint of cream should be added to a gallon of whole milk. Costs £1 (*B.F.J.*, 1930, 98).

Derry. Below the legal (Derry Corporation Act) limit of 5 % of fat to the extent of 37 %, 25 %, 34 % and 35 %, respectively. As they were the first cases, each vendor was ordered to pay 22s. 6d. only (*B.F.J.*, 1931, 106).

YEAST

In 1887 Stock called attention to the adulteration of yeast with potato and other starches. He considered the addition of starch unnecessary for a good yeast, but it made a bad yeast look better. He gave a levigation method and a microscopical method for the determination of the amount of admixture (*S.P.A.*, 1887, 12, 84). R. Williams reported that seven unmixed samples contained 70.0–76.7 % of moisture, and 2.0–3.1 % of ash. A sample containing 21.2 % of starch had 60.8 % moisture and 1.5 % ash (*S.P.A.*, 1888, 13, 86). For the determination of added starch, Dewalque recommends Fehling's solution, after the use of malt extract, followed by inversion (*Analyst*, 1899, 24, 89), and Crispo, polarimetric determination in alkaline solution (*Analyst*, 1900, 25, 39).

Yeast extract has been used as a substitute for meat extract; analyses of various samples of each have been made by Graff (*Analyst*, 1904, 29, 194), and analyses of yeast products by Willimott and Wokes (*Analyst*, 1928, 53, 609), who also compare the vitamin B content (cp. p. 295).

There has been a great diminution of adulteration of yeast examined in England and Wales. In 1891–4, 11 % of the samples were adulterated; in 1905–13 the proportion fell to 1.6 %, and only one out of the 767 samples during 1920–30 was condemned.

PROSECUTIONS. *Pontypridd*. Potato starch 14 %, and dry yeasty substance 20.6 %, instead of 26 %. Fine £2 (*F. & S.*, 1896, Sept. 26, Oct. 24).

Tywardreath. Cornflour 30 %. Fine £1 8s. 6d. (*B.F.J.*, 1904, 46).

Norton. Flour 35 %. After the sale the inspector was shown a notice, "To improve the keeping qualities of the yeast, ours is mixed with a small proportion of pure farina." Paid costs (*Grocer*, 1910, April 30).

TABLE SALT

The chlorine in five Birmingham samples corresponded to 97.4–98.5 % of sodium chloride. Three samples contained 1.3–1.4 % SO_4 , 0.3–0.4 % Ca, and 0.0–0.4 % Mg and traces of iron.

Phosphate was not detected. Salt usually contains potassium chloride, and the presence of calcium and magnesium chlorides makes it slightly deliquescent.

Samples of a prepared table salt, stated to contain phosphates, yielded 1.5–1.9 % PO_4 , 1.3–1.7 % SO_4 , and 1.1 % of Ca.

Standards for sodium chloride have been suggested by the British Committee of the International Union of Pure and Applied Chemistry (*Analyst*, 1924, **49**, 281).

PROSECUTION FOR SALT. *Stradbally*. Carbonates of magnesium and calcium. The addition was stated to have been made to keep the salt dry and to enable it to pour easily. The District Justice dismissed the case on payment of costs, and asked the defendant to take up the matter of labelling with the manufacturers (*Analyst*, 1928, **53**, 383).

OTHER FOODS

The following percentages of adulteration in England and Wales have been reported for the periods given :—

Beverages, fruit juices, and syrups .	1920–30	14.5%
Milk preparations	1920–30	3.0 %
Pickles	1906–13	2.7 %
„	1920–6	1.4 %
Pickles and sauces	1927–30	1.5 %
Soups	1920–30	2.2 %

CHAPTER XXXI

ALKALIS, CARBONATES, SULPHATES

Solutions of ammonia. Lime-water. Fluid magnesia, emulsion of magnesia. Magnesia, magnesium carbonate. Ammonium carbonate. Sodium carbonate and bicarbonate. Potassium bicarbonate. Potassium carbonate, salt of tartar. Sodium sulphate, Glauber's salt. Magnesium sulphate, Epsom salt. Sulphate of iron, dried sulphate of iron.

SOLUTION OF AMMONIA

THE weak solution of ammonia of the B.P. contains about 10 w/w of ammonia (NH_3), and the strong solution about 32.5 w/w; the latter has sp. gr. about 0.888. There is also a commercial article sp. gr. 0.880, which may be as strong as 35 w/w. Prosecutions have taken place because vendors have not observed the distinctions between them. Solutions of ammonia, when sold for household cleansing purposes, are not "drugs."

In some cases deficiencies have been attributed to evaporation. R. A. Cripps made experiments on the rate of loss. Strong solution kept in a 5-oz. stoppered bottle, which was occasionally opened for testing, during three years fell from 32.8 w/w NH_3 , to 31.5 w/w. A solution of ammonia in the same conditions fell from 10.3 w/w to 9.7 w/w, and even when kept in a corked bottle the strength was 9.6 w/w, the cork being somewhat shrunken (*P.J.*, 1907, April 27).

PROSECUTIONS FOR SOLUTION OF AMMONIA. *Evesham*. Strong solution was supplied. Fine 1s. 6d. and £2 3s. 6d. costs (*P.J.*, 1904, May 14).

Evesham. Deficient of 12 % of strength. Fine £2 (*P.J.*, 1908, Sept. 19).

Evesham. "Solution of ammonia, B.P." Strong solution supplied. Fine £2 (*P.J.*, 1910, Aug. 27).

PROSECUTION FOR STRONG SOLUTION OF AMMONIA, B.P. *Oldbury*. Ammonia deficient 4.5 %. The sample was supplied from a bottle, marked "Sp. Gr. 0.880," which had been in stock some months. Fine 1s. (*P.J.*, 1911, June 3).

LIME WATER

The alkalinity of lime water is required by the 1914 B.P. to be equivalent to 0.112 w/v of lime (CaO). In 1906, following a prosecution for weak lime water, there was considerable correspondence in the *Chemist and Druggist* on the subject, and

suggestions were made to enable it to be sold of proper strength. They included—(1) using a lime which has been purified by washing ; (2) care in the storage of it so that it is not converted into carbonate ; (3) renewal of the lime in the bottle when necessary ; (4) allowing a sufficient time for saturation with occasional shaking, two days or more ; (5) avoiding heating, which diminishes its solubility ; and (6) avoiding undue exposure to air. One pharmacist, W. S. Clark, reported that in ten days a sample, from which periodical quantities had been poured, only fell from 0.1239 w/v to 0.1223 w/v of CaO (*C. & D.*, 1906, Sept. 29). With a moderate amount of care, there appears to be little difficulty in supplying lime-water of full B.P. strength.

STRENGTH OF CLEAR LIME WATER, 1913-8 (Sixty-six samples)

Lime, CaO, w/v	0.015	0.057-0.075	0.083-0.099	0.108-	0.12-	0.13-	0.140	0.146	Total.
Percentage of samples	3	8	8	16	35	19	11		100

In addition to the above, eight samples were turbid, due to the presence of chalk ; in six samples it was 0.001-0.006—one had 0.016, and another 0.032 w/v. In seven samples the turbidity was due to suspended lime, the quantities in five samples being 0.004-0.050, while the total lime in the other two was about three times the proper quantity, the *excess* undissolved lime being 0.191 and 0.218 w/v, respectively. A number of vendors of incorrect samples were cautioned, and a considerable improvement in quality resulted.

Lime water should be free from lead ; 95 % of the samples contained none or traces ; the other 5 %, 0.4-0.8 per 100,000.

Of the seventy-six Birmingham samples examined 1913-8, 21 % were condemned either for deficiency or excess of lime. The proportion in England and Wales was 32 % during 1897-1900, and 11 % during 1905-13.

ANALYSIS. Titrate 25 ml. with N/10 HCl, using phenol phthalein for the free lime, and then methyl red for chalk, about 0.004 w/v of which is usually present in a clear sample. The alkalinity of turbid samples should be determined, and then that of the clear part, separated either by filtration or sedimentation. A confirmation of the composition may be obtained by adding ammonium carbonate solution, evaporating to dryness, drying and weighing the carbonated solids. The result should be similar to that obtained by calculating the lime present into chalk. The use of a hard water instead of distilled water will increase the carbonated solids.

PROSECUTIONS FOR LIME WATER. *London, Thames.* Water 85 %. The inspector admitted that the article was liable to decomposition, and the certificate was objected to, as it contained no reference to decomposition. The Public Analyst gave evidence

that any change that had taken place was infinitesimal. The defendant admitted that he had found his lime water to be about half strength. The magistrate decided that the decomposition, referred to by the Act, was such as to interfere with ordinary analysis, and there was no evidence that this had occurred; the analyst would have noted the turbidity. He found that the article to which the certificate referred was substantially the article sold, and fined the defendant 10s. The magistrate refused to state a case for appeal, and the Queen's Bench Appeal Court refused to grant a mandamus to compel him to do so (*C. & D.*, 1898, May 28, June 4, June 11, July 9; *F. & S.*, 1898, 882, 894).

Bromley. "26 % of lime had been extracted." Dismissed, as no proof that the defendant had extracted the lime (*C. & D.*, 1900, Aug. 25).

London, Worship Street. Lime 20 % deficient, and prepared with tap water. Fine £1 (*B.F.J.*, 1902, 20).

Greenwich. 171 % more lime than permitted by B.P. Defendant said his lime water would go three times as far as the ordinary lime water. Fine £3 (*P.J.*, 1904, Jan. 16; *B.F.J.*, 1904, 17).

Peterborough. "0.0786 % of lime (CaO), instead of 0.116 %, being deficient to the extent of 33 % of the amount of lime specified in the B.P." The certificate was objected to; it stated, "I am of opinion that the said sample contained the parts as under," but the parts were not under; it might have been ink, which contained a certain amount of lime. A complete analysis would show the amount of lime present in the form of carbonate, which would indicate if the article was originally B.P. The magistrates dismissed the summons, thinking the certificate was hardly an analysis as required by the Act (*P.J.*, 1906, April 7).

London, Clerkenwell. Lime deficient 92 %. Fine £4 (*P.J.*, 1909, July 17).

London, Clerkenwell. Lime 0.0924 %, instead of 0.1168 %. The deficiency was attributed to insufficient shaking during preparation. Paid £2 costs (*P.J.*, 1917, Aug. 25).

Birmingham. "Lime (CaO) 0.000 gm. per 100 ml., calcium carbonate 0.006 gm. Other solid matter 0.018 gm., and water sufficient to make 100 ml. Lime water should contain the equivalent of rather more than 0.1 gm. of lime (CaO) per 100 ml." The bottle was labelled "Pure lime-water." Fine £3 (1918 Report).

London, Old Street. Lead 2.4 parts per million. The defendant's analyst found 2 parts, and the Government analysts 1.6 parts, of which 1.2 parts were in solution and 0.4 parts on the glass. The latter stated that the bottle was made from lead-free glass. The magistrates were satisfied that the lime water was not pure, and to some extent would injure a child. Paid 5 guineas costs (*P.J.*, 1923, Jan. 13; *Analyst*, 1923, 48, 116; *B.F.J.*, 1923, 16).

FLUID MAGNESIA, EMULSION OF MAGNESIA

Liquor Magnesii Bicarbonatis is required by the 1914 B.P. to yield between 0.8–0.95 w/v of magnesia (MgO) on ignition after evaporation, stated to be equivalent to about 2 w/v of the official magnesium carbonate. It is a solution of magnesium carbonate in carbon dioxide, and, unless carefully kept, deposition will occur through the escape of the gas. In the absence of other bases, titration with N/2 HCl and methyl red will give more accurate results than the official ignition method (Evans Report, 1911).

PROSECUTIONS FOR FLUID MAGNESIA. *North London.* Magnesia deficient 63 %. The defendant stated that the bottle from which it had been sold had been opened several times, and attributed the deficiency to deterioration. Fine £4 (*P.J.*, 1908, July 25).

London, Marlborough Street. Carbonate of magnesia 76 % deficient. The case was dismissed as the certificate contained no report on deterioration (*P.J.*, 1909, Jan. 9).

Dublin. Magnesium oxide (residue), at least 14.4 % deficient. It was labelled "Murray's fluid magnesia," and the case was dismissed as it was a proprietary article (*P.J.*, 1921, June 18).

PROSECUTION FOR EMULSION OF MAGNESIA (B.P. Codex). *Feltham.* The article supplied was more than double strength. Paid costs (*P.J.*, 1911, Nov. 4; *B.F.J.*, 1911, 213).

MAGNESIA, MAGNESIUM CARBONATE

The B.P. gives four preparations : Magnesii Carbonas, Levis, and Ponderosa ; Magnesia, Levis and Ponderosa ; light and heavy carbonates and magnesias, or calcined magnesias. They all have the same dose, and the relative commercial values are about 1.0, 1.4, 2.2, and 3.5.

If the Latin terms are used, preparations complying with the B.P. descriptions should be dispensed, but there has been much discussion as to the meaning of the word "magnesia" when it is unqualified ; and also "magnesium carbonate" may be the light or heavy preparation : the practice appears to vary in different parts of the country. Carbonate, however, must not be supplied as "Calcined magnesia," and if "light" or "heavy" is requested the appropriate article must be supplied.

The following advice was given, in 1915, by the Birmingham Pharmaceutical Association's Council :—

"1. When 'magnesia' is asked for it is possible the customer may require (a) magnesia, B.P., (b) magnesium carbonate, (c) fluid magnesia, or (d) effervescent citrate of magnesia, so called. If one of the last two is needed there is usually little

difficulty in arriving at an understanding of the customer's requirements; but usually the public does not appreciate the difference between magnesia and magnesium carbonate, and if it is understood that the carbonate is required *it should be distinctly labelled as such.*"

"2. It is believed that pharmacists do not properly appreciate the rapidity with which magnesia deteriorates upon exposure to the air, and it is desirable to impress upon them that it is quite unsafe to keep this drug packed ready for sale, either in boxes or paper packages. The cost of bottles being prohibitive for small sales, it is recommended that for such sales magnesia (as distinct from the carbonate) be not kept ready packed, but that it be sold *direct from the bottle.*"

The 1914 B.P. required that neither of the magnesias should lose more than 1 % when heated to dull redness. This requirement was unduly stringent, only three of twelve wholesale samples complying with it—they had 0.7–1.0 %; the other nine lost 1.2–2.2 %. None of the retail samples complied with the limit, the lowest being 1.7 % loss; eight others, apart from obviously deteriorated samples, lost 2.2–5.3 %. Retail samples which may have been kept some time should not lose more than 5 % on ignition, which is the limit suggested for the 1932 B.P.

Magnesia which contains about 1.5 % of moisture does not alter in weight on drying in the water oven about two hours; samples with more moisture lose weight on drying; those with less, gain in weight. A sample of calcined magnesia, which had been wetted and dried in the water oven, gained 39.1 % of its original weight, and 0.6 % less on drying at 150°. The chief product of the exposure of magnesia to air is light carbonate (Liverseege, Bagnall and Lerrigo, *B.P. Conf.*, 1926, 468). The loss of moisture in the water oven may be explained as due to the slow rate of combination of MgO and H_2O to form $\text{Mg}(\text{HO})_2$.

The presence of carbonate in calcined magnesia is due to exposure to air during keeping in unsatisfactory conditions; intentional mixture is very improbable. A sample said to have been kept in a cardboard box for three months contained 10.6 % of CO_2 , and lost 3.9 % on drying; another deteriorated sample had 19.5 % of CO_2 and lost 6.9 % drying. A sample of calcined magnesia gained 7.8 % of CO_2 when exposed to the air for three months (November to February) outside the Laboratory in Birmingham (Elsdon and Hawley, *B.P. Conf.*, 1915, 403).

Heavy magnesia is about three times as dense as the light, and the heavy carbonate nearly four times as dense as the light carbonate. Sp. gr., determined as given below, were—light magnesia and light carbonate 0.08–0.16, heavy magnesia 0.36–0.39, and heavy carbonate

0.43–0.48. A sample of light magnesia that had extra heating had sp. gr. 0.22.

The 1914 B.P. required that either of the carbonates shall yield 42–44 % of ash. The upper limit is to be raised to 45 % in the 1932 B.P. The B.P. Codex, 1923, gives the approximate composition of the light carbonate as 3MgCO_3 , $\text{Mg}(\text{OH})_2$, $3\text{H}_2\text{O}$, which indicates 44.1 % of ash, and the heavy to contain $4\text{H}_2\text{O}$, which corresponds with 42.1 % of ash. Birmingham samples of light carbonate yielded 43.6–44.8 % of ash, and the heavy 42.2–43.2 %.

Light carbonate of magnesia, when dried after wetting and exposure to CO_2 , was practically unaltered in weight. The usual amount lost on drying in the water oven was 1.0–1.9 %; the highest amount was 4.9 %.

Further particulars and analyses of twenty-nine Birmingham samples are given in the paper previously cited.

The B.P. limit for lead in each of the four preparations is 20 parts per million. Half of the thirty-four Birmingham samples did not contain more than five parts, and only four were above 15 parts. A sample of commercial (not B.P.) magnesia had 35 parts of lead per million.

None of the thirty-nine samples exceeded the B.P. limit of 5 parts of arsenic per million, and only two of them had over 3 parts.

The twenty-six Birmingham samples of “magnesia” and “calcined magnesia” examined, 1894–1925, gave evidence of carelessness in keeping. Of them, thirteen were more or less deteriorated; only nine yielded 94.7 % or more of ash, while in four instances carbonate had been substituted. There were, however, five duplicates in the unsatisfactory samples.

In England and Wales, 34.5 % of samples of “magnesia” analysed, 1898–1904, were reported adulterated. The description was then altered to “magnesia and preparations”; of these, 17.6 % were condemned during 1905–13, and 18.1 % during 1920–30.

ANALYSIS. The sp. gr. was determined by crushing the sample, when necessary, and putting 20 ml. into a weighed cylinder about 18 mm. in diameter, and weighing again. The cylinder was not shaken except to level the surface at the finish. It is better not to attempt to get more in by tapping.

Besides the determination of the ash, a sample should be examined for loss in the water oven (previously wetting with spirit to avoid mechanical loss), and for the amount of CO_2 . Occasionally the amount of SO_3 , CaO , and Fe_2O_3 with Al_2O_3 is required.

Multiplication of the amount of CO_2 by 0.55, will give the amount of combined H_2O present; this added to the amount of CO_2 and ash and loss on drying should approximate to 100 %. An example may be given. The CO_2 was 10.9 %, and $10.9 \times 0.55 = 6.0$.

The ash was 78.1 % and the loss on drying in the water oven 5.3 %. Total $6.0 + 10.9 + 78.1 + 5.3 = 100.3$. On the basis of 36 % of CO_2 in light carbonate of magnesia, the amount of carbonate in the sample $= \frac{10.9 \times 100}{36} = 30 \%$.

PROSECUTIONS FOR CALCINED MAGNESIA. *Birmingham.* Heavy carbonate of magnesia. Fine £1 (1925 Report).

Dewsbury. Magnesium oxide only 44.5 %, instead of 99 %. The article had been packed in cardboard, allowing deterioration. The summons was withdrawn on a promise being made to improve the packing (*P.J.*, 1925, July 11).

Glasgow. Lime 3.72 %, instead of "the slightest reaction" required by the B.P. The defendant's analyst found 3.40 % of lime, and the Government analysts 3.32 %. The magistrate dismissed the case, holding that the B.P. gave no definite limit, and therefore it was impossible to find that an excess was present (*P.J.*, 1922, April 1; *B.F.J.*, 1922, 36).

PROSECUTIONS FOR MAGNESIA. *Woolton Bassett.* Carbonate of magnesia. Fine 5s. and 7s. costs (*F. & S.*, 1899, Sept. 30).

Leeds. Carbonate of magnesia 100 %. For the defence it was submitted that it had been the custom of the trade for a considerable number of years to recognise carbonate of magnesia as magnesia. On this ground the stipendiary dismissed the case (*C. & D.*, 1900, Sept. 22; *B.F.J.*, 1900, 291).

Ashby-de-la-Zouch. A mixture of magnesia and French chalk, used for pill coating, had been sold by mistake. Fine 2s. 6d. (*B.F.J.*, 1900, 291).

Gateshead. Magnesia 42.25 %, combined water and carbonic acid 57.75 %. The Bench dismissed the case, as the quality of magnesia had not been specified when the purchase was made, and the article was pure of its kind (*B.F.J.*, 1900, 330).

Wolverhampton. Applying a false trade description, "Pure Magnesia," to magnesium carbonate. Fine £5 (*P.J.*, 1903, Oct. 24).

Loughborough. Citrate of magnesia. There was a label, "Citrate of magnesia," on the bottle, which the purchaser did not see. Fine 10s. (*P.J.*, 1916, Oct. 7).

PROSECUTION FOR LIGHT MAGNESIA. *London, Guildhall.* Carbonic acid 11.5 %, water 7.7 %. Fine £5 (*P.J.*, 1907, June 15).

PROSECUTIONS FOR HEAVY MAGNESIA. *Belper.* Light magnesia. Fine 5s. (*B.F.J.*, 1900, 197).

Portsmouth. Heavy carbonate of magnesia 13 %. The sample, which was wrapped in paper, was kept by the inspector three hours, and the Public Analyst allowed the paper packet to lie on his table till he analysed it. The defence suggested that the sample had absorbed moisture between purchase and analysis, and that it should have been placed in a well-stoppered bottle. The case was dismissed,

as the Bench did not think the sample was properly preserved by the inspector and the Public Analyst (*B.F.J.*, 1901, 29).

London, Lambeth. Hydrated carbonate of magnesia 30 %, and moisture 2.7 %, dispensed for a prescription which ordered "Magnesia ponderosa." The defendant had had it in stock at least ten years; evidence was given that the altered article would be equally good for medicinal purposes. Case dismissed (*P.J.*, 1901, March 23; *B.F.J.*, 1901, 138).

London, Islington. Nine chemists dispensed magnesium carbonate for a prescription which ordered "Magnesia ponderosa," tincture of cardamoms and distilled water. Four defendants, who had been previously convicted, were each fined £5 5s., and the other five £1 1s. each (*P.J.*, 1928, March 17).

AMMONIUM CARBONATE

In 1870, E. Divers made a detailed investigation of the carbonate of ammonia of commerce (*J.C.S.*, 1870, 171-279). He found the samples had the composition of one molecule ammonium carbamate with one molecule of ammonium bicarbonate ($\text{N}_3\text{H}_{11}\text{C}_2\text{O}_5$), with not more than 2.5 % of additional water, and a slight excess of NH_3 . He found carbonic anhydride 56.05-54.65 %, and ammonia 33.05-31.67 %.

The 1885 B.P. required 1 gm. to neutralise 19.1 ml. of normal acid (= 32.5 % NH_3); that of 1898 reduced the requirement to "at least 18.7 ml." (= 31.8 % NH_3), and the 1914 edition again reduced the quantity to "at least 18 ml." (= 30.6 % NH_3). The latter result was due to the recommendation of Greenish and Smith (*P.J.*, 1901, 775). Self and Corfield (*P.J.*, 1926, Feb. 6) stated that the B.P. standard was not unreasonable, since resublimed ammonium carbonate frequently contains 4-6 % more than the 18.0 ml. required. The limits suggested for the 1932 B.P. are 30-32.5 % NH_3 . Six samples of "Ammonium carbonate" bought in Birmingham varied 29.5-31.3 % NH_3 . Two lumps of one sample differed by 1.4 % NH_3 , and by 1.8 % in another. The analysis was made by weighing about 1 gm., adding it to water and 20 ml. N.HCl , boiling and titrating back.

On exposure to air, ammonia is lost and the lumps become coated with ammonium bicarbonate, which should be scraped off before using the article for dispensing. Loss of this kind is greater in the powdered salt. Cripps and Hoyland (*P.J.*, 1928, Sept. 8) found that a sample of the powder lost 10 % of its alkalinity, and also 10 % of its weight on exposure to air for one hour. Ammonium carbamate is much more alkaline than ammonium bicarbonate; of the former, 1 gm. = 25.6 ml. N.HCl , and of the latter, 12.7 ml.

Owing to the hardness of the salt and to its proneness to

decompose, it is better to use a solution of the salt for dispensing purposes. Both Self and Corfield (*P.J.*, 1926, Feb. 6) and Cripps and Hoyland (*opus cit.*) have proved that solutions of ammonium carbonate keep well. They also found no appreciable loss of ammonia from dispensed medicines after a month or more. The writer found similar results with a senega mixture (see p. 548).

Ammonium carbonate as sold retail is usually required for cleansing purposes, and is therefore neither a food nor a drug, unless intimation is given on purchasing. W. Johnson (*C. & D.*, 1904, July 30) has recorded experiments in which he found that the bicarbonate was as good a cleanser as the carbonate, and more efficacious for baking purposes.

PROSECUTIONS FOR AMMONIUM CARBONATE. *Great Malvern.* Required only 13 ml. of sulphuric acid solution to neutralise 1 gm., instead of 18·7 ml. Several pharmacists gave evidence that the article was never sold for medicinal purposes. The case was dismissed, the magistrates considering that the article was not purchased as a drug, and there was no standard for the commercial article (*C. & D.*, 1904, July 2).

Oldbury. Deficient in strength 16·05 %. On a slip of paper handed to the chemist was "4 oz. of ammonium carbonate, B.P." Paid costs (*P.J.*, 1914, Dec. 19).

SODIUM CARBONATE, SODIUM BICARBONATE

In popular usage the above terms are synonymous, true sodium carbonate ($\text{Na}_2\text{CO}_3, 10\text{H}_2\text{O}$) being called "Soda" or "Washing soda." Although the latter is in the B.P., and is used as an external application for rheumatism, it cannot be assumed that when sold it is a "drug," unless some intimation to that effect is given at the time of purchase.

Sodium bicarbonate is rarely adulterated; of 151 Birmingham samples, bought 1890–1929, two only, from one vendor, were incorrect, being borax. This accidental substitution had a disastrous effect on some pastry; the vendor was cautioned, on promising to advertise in the newspapers that a mistake had occurred, and to compensate anyone who had suffered by the mistake. The substitution probably arose from the resemblance between "Sod. Bic." and "Sod. Bib.," which is borax.

The B.P. gives a lead limit of 5 parts per million; 6 % of eighty-nine Birmingham samples had 6 or 7 parts, 30 % 3–5 parts, and 63 % 0–2 parts. Arsenic has not been detected. During 1905–13, 2·8 % of the samples examined in England and Wales were adulterated, and 3·7 % during 1920–30.

PROSECUTION FOR BICARBONATE OF SODA. *Llandrindod Wells.* Arsenic, 100 parts per million, and borax were present. It

was stated that the article had been accidentally mixed with commercial borax. Fine £2, and the assistant 7s. 6d. costs (*P.J.*, 1920, Dec. 26).

PROSECUTIONS FOR CARBONATE OF SODA. *Ripley.* Bicarbonate of soda. When the purchase was made the inspector did not mention "B.P." Evidence was given for the defence that twenty-eight London chemists each supplied bicarbonate on being asked for "carbonate." Case dismissed (*P.J.*, 1901, June 29; *B.F.J.*, 1901, 243).

Driffeld. Crystallised borax 75.7 %. Fine 15s. (*Grocer*, 1930, Jan. 1; *B.F.J.*, 1930, 15).

PROSECUTION FOR SODA. *Marylebone.* Glauber's salt 42 %, a "food," as it was used in the boiling of vegetables. The magistrate dismissed the case, holding that its use for retaining the colouring of greens did not make it a food, and that it could not be said to "enter into the composition or preparation of food." Five guineas costs were allowed to the defendant. Five other summonses against vendors, whose articles contained 43, 50, 51, 68, and 70 %, respectively, of Glauber's salt, were withdrawn (*P.J.*, 1904, Nov. 26, Dec. 24; *B.F.J.*, 1905, 19).

PROSECUTION FOR WASHING SODA. *Wandsworth.* Applying a false trade description to an article containing 42–45 % of sulphate of soda (Glauber's salt). When the vendor was told it was for analysis, he said, "It is a mixed soda." For the defence it was argued that the Merchandise Marks Act referred only to marks applied to goods, and there had only been a verbal request. The magistrate ruled that the word "description," used in the Act, was meant to cover a verbal as well as a written description, and fined the defendant £4 (*P.J.*, 1904, Sept. 16).

PROSECUTION FOR SODA CRYSTALS. *Tunbridge Wells.* Applying a false trade description, "Soda crystals," to an article containing 34 % of Glauber's salt. Evidence was given that the article was much cheaper than ordinary soda, which contained only 1–2 % of sulphate of soda. The magistrates decided that "Soda crystals" were washing soda, and that a false description had been used. Nominal fine of 10s. (*Grocer*, 1904, Dec. 17).

POTASSIUM BICARBONATE

Only three of the 106 Birmingham samples examined 1890–1926 were condemned. Two of them were the much stronger potassium carbonate, and three-quarters of the other was sodium bicarbonate. Three of the seventy-nine samples examined 1916–26 exceeded 2 parts of arsenic per million, having 3–5 parts. The lead in 40 % of these samples was 0–2 parts per million; in 46 % it was 3–5 parts, and 14 % slightly exceeded the B.P. limit of 5 parts, having 6–9 parts.

POTASSIUM CARBONATE

The 1898 B.P. introduced "Salt of tartar" as a synonym, but the 1914 edition omitted the synonym, and it would appear better to retain that name for the commercial article, which may contain 60–100 parts of arsenic per million. In Germany, its presence has been attributed to arsenical sheep dips contaminating the wool from which potassium carbonate is prepared (*Analyst*, 1925, **50**, 197).

The B.P. (1914) limits for arsenic and lead are 5 parts and 2 parts per million, respectively, and at least 81.5 % should be K_2CO_3 , while the moisture lost on ignition should not exceed 18.5 %, the ignited residue being therefore at least 81.5 %. The following results were obtained with eighteen Birmingham samples bought in 1923 and 1926. Three, of satisfactory quality, were labelled "Salt of tartar," the others "Carbonate of potash" :.

ANALYSES OF POTASSIUM CARBONATE

Arsenic, parts per million	. 0	1	4	7	10	Total.
No. of samples	. 8	6	2	1	1	18
Lead, parts per million	. 0, 1	2, 3	8, 10	80	270	Total.
No. of samples	. 6	5	4	2	1	18
K_2CO_3 , or ignited residue, %	. 76.1-79.4	80.0-81.5	81.6-82.9	83.9-90.7		Total.
K_2CO_3 , No. of samples	. 3	7	5	3		18
Ignited residue, No. of samples	3	4	8	3		18

The appearance of the samples was not a sure guide as to the amount of moisture present. Each of the three samples having more than 20 % of moisture appeared slightly damp, but only about half of those having 20–22 %.

Three samples contained the remarkable amounts of 270 and 80 parts, respectively, of lead per million, and no other impurity, except that the first sample had 5 % of water over the limit. The two samples having 80 parts were from one vendor and had 7 and 10 parts, respectively, of arsenic per million ; they did not appear damp.

The vendor was prosecuted, and it was proved that the potassium carbonate contained no excess of lead or arsenic when it was put into the shop bottles. Before 1923 no suggestion had been made that *dry* potassium carbonate would attack glass, but experiments made for the defence proved lead and arsenic might be removed from the glass bottle (Richmond, *S.P.A.*, 1923, **48**, 260).

The writer made severe tests by mixing ground glass with an equal quantity of B.P. potassium carbonate, and keeping in a corked bottle for three months. The soluble lead was then found to be about 1,400 parts per million, and the arsenic 70–100 parts per million. It is probable that the variations in temperature in a pharmacy will produce condensation of moisture on the sides of a

bottle containing potassium carbonate, and that the damp salt thus produced will attack the glass in contact with it. It is obvious that glass bottles used for storing potassium carbonate should be free from lead and arsenic (Liverseege, *Analyst*, 1923, **48**, 543).

PROSECUTIONS FOR SALT OF TARTAR. *Wolverhampton.* Arsenic 0.14 %. The purchase followed a prosecution for arsenic in beer, due to the addition of salt of tartar to make it palatable. The vendor had labelled the salt of tartar "Poison," expecting it to be used only as a hair wash. Fine £2 (*P.J.*, 1914, April 18; *B.F.J.*, 1914, 80).

Manchester. Arsenic 4.5-9.5 grains per lb., from five chemists. For the defence it was argued that the article was neither a food nor a drug, and that its use in whooping-cough mixtures was obsolete. The prosecutions were withdrawn, on payment of costs, on the defendants undertaking to warn purchasers of the commercial article, by a label, that it was not for internal use (*P.J.*, 1914, June 20; *B.F.J.*, 1914, 138).

Burslem. Arsenious oxide 1.4 grains per lb., which was attributed to the use of impure sulphuric acid in its manufacture. The stipendiary considered the article was bought as a drug, and that the unexplained presence of this quantity of arsenic was sufficient to make the sale to the prejudice of the purchaser. Fine £2 (*B.F.J.*, 1914, 160).

Birmingham. Lead about 80, and arsenic about 10, parts per million. Evidence was given that the article was pure when put into the shop bottle, which contained arsenic and lead, and that the impurity was due to the bottle. The case was dismissed on the ground that the drug was "unavoidably mixed with some extraneous matter in the process of collection or preparation," and that "collection" included storage (*Analyst*, 1923, **48**, 215).

SODIUM SULPHATE, GLAUBER'S SALT

Of the 186 Birmingham samples examined 1912-29, two samples from one vendor consisted of Epsom salt, probably due to accidental substitution. The B.P. gives limits of 5 and 2 per million, respectively, for lead and arsenic.

LEAD AND ARSENIC IN GLAUBER'S SALT

Parts per million, lead, or arsenic	0-2	3-5	6-12	35, 40	Total.
PERCENTAGE OF SAMPLES:					
Lead, 1912-9	65	20	15	—	100
„ 1920-9	87	12	1	—	100
Arsenic, 1912-9	88	3	7	2	100
„ 1920-9	99	1	—	—	100

The more unsatisfactory results in the first period were probably due to the difficulties of war-time conditions. Six samples were

tested for sodium nitrite, and 0·2 parts per million were found. Chlorine was determined in eleven samples; the largest amount corresponded to 0·3 % of sodium chloride.

Of the samples examined in England and Wales during 1920–3, 3·4 % were reported adulterated, but later samples (1924–9) were genuine, while four out of the 149 examined in 1930 were adulterated.

PROSECUTIONS. *Birmingham.* Arsenic 8 and 10 parts per million, respectively. On behalf of the two vendors it was stated that the manufacturer who supplied them had not properly purified it owing to war conditions. The prosecutions were withdrawn on the vendors undertaking to supply the pure article in the future (1916 Report).

Moneymore. Lead 20 parts per million. The defence was that the salts were for animal use, and the case was dismissed (*Grocer*, 1930, Oct. 18).

MAGNESIUM SULPHATE, EPSOM SALT

The 1914 B.P. prescribed limits of 5 parts per million for lead and arsenic. About 100 samples were examined in Birmingham during 1915–29. Only one sample exceeded the arsenic limit, 20 parts being present; it was a war-time sample, marked “purified.” In 91 samples the arsenic was either 0 or 1 part. In the 1932 B.P. the arsenic limit is to be reduced to 2 parts. The amount of lead in five samples exceeded the limit; four, war-time samples, had 8–10 parts, while ninety-two samples had 0·2 parts of lead per million.

The small crystals of magnesium sulphate are very similar in appearance to those of zinc sulphate, and occasionally the two salts get mixed with disastrous results, examples of which are mentioned in the L.G.B. Reports for 1907 and 1911. Owing to wrong labelling, a sample of zinc sulphate was sold as Epsom salt in Birmingham, but fortunately the mistake was discovered before anyone was injured.

During 1905–13, 3·9 % of the samples examined in England and Wales were adulterated, and 1·0 % of those during 1920–30.

PROSECUTIONS. Arsenic $\frac{3}{4}$ and 1 grain per lb., respectively. Impure materials had been used in their manufacture. Fines and costs £3 5s. and £5, respectively (L.G.B. Report, 1913).

Draperstown. Arsenic 80 parts per million. The grocer who sold it was fined 1s. (*B.F.J.*, 1916, 372).

Walthamstow. Arsenic and lead, due to impure acids. Fine £1 (*B.F.J.*, 1918, 18).

Manchester. Arsenic 1 gr. per lb. The commercial quality had been sold instead of the medical. Fine £1 (*B.F.J.*, 1918, 66).

Neston. Arsenic 50 parts per million. The grocer who sold it was fined 10s. (*Grocer*, 1919, Oct. 11).

SULPHATE OF IRON, DRIED SULPHATE OF IRON

The titration by permanganate of nine Birmingham samples of pure sulphate of iron indicated 100.3–101.5 % of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, indicating that a little moisture had been lost. Another sample indicated 99.4 %. On drying at 130° C. they lost 37.6–39.1 %, corresponding to about 6 molecules of water. The loss in the water oven was usually about 0.5 % less. On exposure to air on watch glasses about 19 % was lost, equivalent to about 3 molecules of water. The weight became constant in about twelve days. One oz. kept in an open wide-mouth bottle lost 1 % in two weeks, and 5.6 % in fourteen weeks. At the end of that time part of the sample was white. With reasonable care in keeping, the increase of strength by loss of water is trifling. Arsenic was not detected.

The 1914 B.P. requires dried sulphate of iron to contain at least 77 % of FeSO_4 ; in the 1932 B.P. the limit is to be raised to 80 %. Twelve Birmingham samples indicated 69.5–84.1 %. The loss on drying in the water oven was 2.3–19.8 %, figures which indicate a very variable substance. About 0.5 % more was lost on drying at 130° C. Seven of the samples were below the B.P. limit, chiefly owing to the amount of moisture present. If the moisture had been removed by drying, all the samples would have passed the B.P. limit. The B.P. also requires slow but entire solubility in water. The amount insoluble varied 0.3–3.3 %. It was determined by treating with boiled cold water in a closed flask completely filled, the insoluble matter being dried and weighed. The arsenic varied 0–5 parts per million. In the 1932 B.P. the limit for arsenic is to be altered from 5 parts to 2 parts per million.

Papers on this drug have been given by Lunan (*P.J.*, 1888, 226), Lothian (*P.J.*, 1898, 668), Cowley and Catford (*C. & D.*, 1900, Schl. 475), Liverseege (*B.P.C.* 1924, 757; *P.J.*, 1927, Jan. 29), and Abraham (*P.J.*, 1927, Dec. 17).

CHAPTER XXXII

NATURAL DRUG PRODUCTS

Rhubarb. Powdered gentian root. Ground liquorice root. Senna leaves. Saffron.

RHUBARB

THE 1914 B.P. gave a limit of 15 % for ash. The writer has shown, however (*P.J.*, 1922, May 20), that, owing to the amount of calcium oxalate in the drug, the ash is an indefinite figure, one sample giving 11.5–19.3 % by varying the conditions of ashing. The carbonated ash is a much more definite figure.

ASH AND CARBONATED ASH OF POWDERED RHUBARB

AMOUNT, %	6.9–	8–	10–	12–	14–15.2	Total.
PERCENTAGE OF SAMPLES :						
Ash	8	28	41	23	—	100
Carbonated ash	—	14	31	41	14	100

Four samples of “East India” lump rhubarb gave 11.4–29.7 % of ash, and three samples of English lump 7.7–12.0 %.

The amount of moisture in twenty-nine samples of powder varied 5.6–8.9 %. Detailed analyses of twelve samples of powdered rhubarb have been given by Liverseege, Bagnall and Lerrigo (*B.P. Conf.*, 1926, 465). The amount of cold water extract varied 37.1–47.4 %, the methylated spirit extract 33.8–42.6 %, and the amount insoluble in E/2 acetic acid 46.2–57.2 %.

ANALYSIS. The method of determination of carbonated ash has previously been given (p. 71), and that for amount insoluble in acetic acid is given under “Gregory’s Powder” (p. 481). Determine the cold water extract by shaking 0.75 gm. with 150 ml. of water, filtering the next day, and evaporating and drying 100 ml. of the filtrate. For the methylated spirit extract treat 0.7 gm. of rhubarb similarly with 70 ml. industrial methylated spirit, and evaporate and dry 50 ml.

Emodin may be shown to be present by boiling 0.1 gm. with 10 ml. E/5 KOH for a few minutes. Then cool, filter, acidify with HCl, shake out with 10 ml. ether, separate the ethereal layer, and a cherry-red ethereal layer should appear on shaking with 6E. AmOH. Methods for its determination have been given by Valaer (*Analyst*, 1931, 56, 817).

Turmeric may be detected by pouring about 7 ml. of hot methylated spirit on about 0.5 gm. placed in the apex of a 7-cm.

filter. Heat filtrate in basin with solid boric acid and a few drops of HCl with H_2O_2 solution. If 1 % of turmeric be present, there will be a red deposit on the sides of the basin.

PROSECUTION. *London, Thames.* Foreign starch, resembling maize starch, at least 20 %. Summons adjourned (*P.J.*, 1909, Feb. 13).

GENTIAN ROOT

In 1904, Collins (*C. & D.*, 1904, March 5; *Y.B.P.*, 1904, 214) called attention to the adulteration of this root with powdered almond shell. He removed the gentian by levigation and examined the heavier deposit microscopically. One sample was largely coloured pinewood and silicious matter, and had 9.0 % of ash. Bell (*P.J.*, 1908, Aug. 29) found that after shaking with water some time, an adulterated sample gave a less bulky deposit than a genuine one. In 1905 there were a number of prosecutions for large proportions of ground olive stones (*poivrette*), said to have been mixed in the South of France, and whole stocks were called in and burnt. Greenish and Bartlett (*P.J.*, 1912, Feb. 17; *Y.B.P.*, 1912, 252) examined thirty-three samples; ten of them were adulterated with almond shells or woody matter. Some of these samples yielded only 10.6–13.0 % of soluble matter to water, instead of 33 %, which could be easily attained. These workers suggested 5 % of ash as a limit. A sample examined by Bruchhausen (*Y.B.P.*, 1913, 271) contained powdered coconut shell. The B.P. requires at least 33 % of water-soluble matter, and not more than 6 % of ash. Of the 113 samples examined in England and Wales during 1905–7, twenty-nine were adulterated.

PROSECUTIONS. *Consett, Durham.* Ground olive stones 25 %. It was said to be used for agricultural or veterinary purposes. Fine 10s. and costs (*P.J.*, 1905, Oct. 28).

Chester-le-Street. Ground olive stones 35 %. Two vendors were each fined 5s. and costs (*P.J.*, 1905, Dec. 9).

Guildford. Ground olive stones 20 %. For the defence it was argued that the summons was for selling a "drug," and that no evidence had been given that powdered gentian root was a drug. Evidence was given that it was only given to animals and never to human beings. The Bench held that it was a "drug" within the meaning of the Act, and fined the defendant £2 (*P.J.*, 1907, March 16).

Brighouse. Ground nut shells 50 %. The defendant stated that the drug had been ground in Marseilles, and that he was unaware of the adulteration; he was ordered to pay costs only, 28s. (*P.J.*, 1907, May 18).

Penkridge. Foreign ingredients, probably sawdust, 20 %. As the article had been destroyed, the vendor was only ordered to pay £1 (*P.J.*, 1907, Nov. 2).

Old Hill, Wolverhampton. Ground almond shells 25 %. The article had been destroyed, and the defendant was ordered to pay costs only (*P.J.*, 1907, Nov. 2).

Manchester. Almond shell 75 %. Fine £10 (*P.J.*, 1931, March 28).

Bishop Auckland. Starch and other matters 30 %. Fine £2 (*P.J.*, 1931, May 9; *B.F.J.*, 1931, 65).

GROUND LIQUORICE ROOT

The 1914 B.P. requires the decorticated root to be used; it describes the powder as yellow, and gives an ash limit of 6 %. Greenish and Bartlett (*P.J.*, 1913, March 15) examined nine samples of commercial root, and found 3.3–7.8 % of ash in the dried root. In thirty-two samples of commercial powder, some of which were adulterated, the ash varied 3.8–7.4 %. The ash of twelve samples of yellow powder and decorticated root examined in Birmingham varied 3.4–5.5 %; another had 6.4 %; the ash insoluble in acid was only 0.1–0.5 %. The cold-water extract of these samples varied 24.8–34.2 %. The determination was on 2 gm. macerated in 100 ml. two days. In the 1914 B.P. test the proportion is five times as great, and chloroform water is used for twenty-four hours. In several tests the B.P. method removed much less soluble matter than the weaker mixture. The fifteen samples described by Greenish and Bartlett as “normal” yielded 25.0–38.4 % of soluble extract, the proportion used being half that of the B.P.

Grinding the undecorticated root yields an inferior brownish powder. The ash of eight Birmingham samples of these roots and powders was 4.2–8.6 %, the sandy matter 0.1–2.5 %, and the cold-water extract 20.1–28.0 %. Another sample had 14.4 % of ash, 9.0 % being insoluble in acid. It had 35.5 % of crude fibre, and was almost worthless, only 5.6 % being soluble in water. A prosecution was instituted, but the summons was dismissed! While the Food and Drugs Act limits “food” and “drink” to those “used by man,” there is no such limit to “drugs.”

MICROSCOPICAL EXAMINATION. The structures present are described by Scott-Smith and Evans (*S.P.A.*, 1911, **36**, 198), and a plate is given. Ground olive stones have been detected.

PROSECUTIONS. *Gateshead.* Cornflour 10 %. Fine 10s. (*P.J.*, 1905, Aug. 19).

Liverpool. Ground olive stones 40 %. The powder was said to be a dark one imported from Marseilles for veterinary use. Fine £10 (*P.J.*, 1905, May 13).

Birmingham. Ash 14 %, soluble in water 6 %, while ground liquorice root should not contain more than 9 % of ash, and at least 16 % of it should be soluble in water. The defence was that the

powder was the dark one used for veterinary purposes, but the counsel did not argue that it was not a drug. The magistrates considered the case was not proved, and dismissed it, but declined to award costs to the defendant (*P.J.*, also *C. & D.*, 1919, March 22).

SENNA LEAVES

The B.P. of 1914 recognises both Alexandrian and Indian (Tinnevelli) senna, and requires that the ash shall not exceed 12 %. A description of the microscopical structure of the leaf, with a plate, has been given by Scott-Smith and Evans (*S.P.A.*, 1911, **36**, 201). Heisch (*S.P.A.*, 1888, **13**, 150) and Parkes and Major (*S.P.A.*, 1914, **39**, 162), have given detailed analytical figures of both kinds.

Five of the nineteen Birmingham samples of leaves contained an undesirable proportion of small stones and grit not adherent to the leaves, the range being as follows :—0.0.2 %, eight samples ; 0.3–0.5 %, six samples ; 1.6–3.5 %, five samples.

The ash of powdered senna is higher than that of the leaves, after the removal of the loose grit.

ASH IN SENNA LEAVES AND POWDER

Ash, %	8.4–9.2	9.4–10.4	11.2–11.9	12.1–13.4	Total samples.
Senna leaves	5	14	2	0	21
Powdered senna	0	0	2	6	8

Greenish (*P.J.*, 1901, March 30) attributes the larger proportion of ash in the powder to the removal of ungrindable residue (“gruff”), which, however, would not amount to 7 % of the drug. The powder, containing the tiny crystals of calcium oxalate, was about 2 % higher in ash than the gruff. He also gives the ash of a number of varieties of senna.

The ash of the leaves, after removal of loose grit, was treated with dilute HCl and the ash insoluble in acid weighed. Eight samples had 0.0.4 %, and thirteen samples had 0.5–0.8 %. Four samples of powder had 1.3–2.9 % of insoluble ash, and four had 3.0–4.0 %, figures which suggest that loose grit had been included in the powders.

MOISTURE IN SENNA LEAVES

Moisture, %	6.9–	8.0–	9.0–10.0	Total samples.
Samples	6	6	8	20

Treatment with cold methylated spirit (4 w/v), for two days, and weighing the soluble extract, indicated that the powders yielded more than the leaves ; some of the latter were considered to be of inferior quality :—

SPIRIT EXTRACT OF LEAVES AND POWDER

Spirit extract, %.	6.2-8.9	9.5-10.7	10.9-12.2	12.6-14.3	Total samples.
Senna leaves	4	7	4	0	15
Powdered senna	0	0	3	5	8

In all the above figures there was very little difference between the two varieties of leaves ; samples of powdered senna are included in the following figures, as they also are similar :—

COLD-WATER EXTRACT OF SENNA (2 W/v)

Cold water extract, %	27.4-30.4	31.1-34.8	35.6-36.6	Total samples.
Leaves and powder	4	20	5	29

Powdered senna is one of the ingredients of compound liquorice powder, and the amount of sulphate, expressed as sulphur, was determined in eight samples ; in some it was absent, and the highest was 0.05 %. The carbon-disulphide extract of one sample was 6.1 %, and the fibre in it 10.0 %.

SAFFRON

There are three varieties of saffron :—(1) Valentia, or hay saffron ; (2) Alicante saffron, which is often grossly adulterated with mineral matter ; and (3) cake saffron, which consists of safflower florets made into a cake with sugary or gummy matter, and the cost may be only about one-twentieth of that of genuine saffron. The first only was recognised in the 1898 B.P., which gave limits of 7.0 % for ash and 12.5 % for moisture. It was omitted from the 1914 B.P. as being of little medicinal value, but is still in popular demand for making a drink in cases of measles, etc., and therefore samples should be taken. It is an expensive drug and has been largely adulterated.

References may be made to papers by Maisch (*Analyst*, 1885, **10**, 200), Barclay (*C. & D.*, 1894, Feb. 24), Parkes (*P.J.*, 1908, Feb. 29), Bulir (*Analyst*, 1913, **38**, 420), Krzizan (*Analyst*, 1914, **39**, 312), and those by Pierlot (*Analyst*, 1916, **41**, 278 ; 1923, **48**, 389 ; 1926, **51**, 41).

Of the samples examined in England and Wales during 1908-13, 27 % were adulterated. In 1889-93, seven of the twenty-nine samples examined in Birmingham were adulterated, and several convictions ensued. During 1894-1915, only two of the forty-one samples were condemned.

ASH IN SAFFRON

Ash, %	4.0	4.5-	5.0-	5.5-	6.0-	6.5-7.1	10-35	Total.
No. of samples	1	16	17	12	8	9	5	68

The five samples containing over 10 % of ash were obviously

adulterated, and the one with 4.0 % may have contained an organic adulterant.

MOISTURE IN SAFFRON

Moisture, % . . .	6.9–	9–	10–	11–	12–	13, 14	16–18	Total.
No. of samples . . .	12	5	10	7	6	4	3	47

Several samples exceeded the B.P. limit. On treating seven genuine samples with water, 52.4–55.9 % of soluble matter was extracted, which included 3.8–6.1 % of ash.

ANALYSIS. If saffron be sprinkled on warm water vegetable substitutes, such as calendula florets, become obvious, and also saffron stamens, which may be present up to about 2 % in genuine samples. Every particle of genuine saffron should give a blue colour with B.P. H_2SO_4 . Calendula florets turn brown or blackish brown, and those of carthamus, yellow (Kraemer). A good sample of saffron should have about twenty-five times the tinctorial power of $\text{K}_2\text{Cr}_2\text{O}_7$ (Dott).

There should be no deflagration on ignition, and a low ash may be due to the presence of sugar, etc. The determination of nitrogen may sometimes be useful; 2.2–2.4 % should be present.

PROSECUTIONS. Savin was supplied instead of saffron. As the savin was unadulterated, the magistrates dismissed the case. On appeal, *Knight v. Bowers* (1885), it was decided that an offence had been committed.

Birmingham. Dyed calendula florets 55 %, mineral matter 25 % (28 % of the sample was sandy matter). Fine £3 (1889 Report).

Birmingham. Foreign vegetable and mineral matter 70 % (the sample contained 43 % of dyed calendula florets and yielded 35.1 % of ash). Fine £3 (1892 Report).

Birmingham. Foreign vegetable matter (sedge) 60 %, colourless saffron 10 %. Fine £1 (1892 Report).

Castleford. Sugar 75 %, safflower 25 %, and known as “cake saffron.” Case dismissed, as particulars of the offence were not given in the summons (*C. & D.*, 1892, Dec. 17).

London, Clerkenwell. Barium sulphate 36 %. Sold by a tobaccoconist, who was ordered to pay costs (*F. & S.*, 1897, March 20).

London, Clerkenwell. Saltpetre 25 %. Costs (*P.J.*, 1907, Oct. 26; *B.F.J.*, 1907, 214).

Bilston. Borax 4.75 %, potassium nitrate 8.3 %. Case dismissed as warranty proved. Epsom salt 5.1 %, sodium borate 5.6 %. Fine 10s. (*P.J.*, 1910, July 16; *B.F.J.*, 1910, 179).

London, Old Street. Borax 37 %. The article was said to be Alicante. Fine £2 (*P.J.*, 1914, Feb. 7).

Penzance. Moisture 23.1 %, being above the limit of 12.5 %. Fine 5s. (*P.J.*, 1926, Feb. 13; *B.F.J.*, 1926, 29).

Camelford. Added mineral salts 80.3 %, excess moisture 10.7 %. Dismissed on warranty. The wholesale dealer was subsequently fined £5 for false warranty (*P.J.*, 1927, Jan. 22, March 5).

CHAPTER XXXIII

MEDICINAL POWDERS

Pepsin. Wood charcoal. Saccharin, gluside. Precipitated sulphur, milk of sulphur. Reduced iron. Oxide of iron. Sodium phosphate. Mercury with chalk, grey powder. Compound liquorice powder. Compound powder of cinnamon. Compound rhubarb powder. Gregory's powder

PEPSIN

THE 1914 B.P. requires that pepsin should dissolve 2,500 times its weight of coagulated white of egg in specified conditions. Cameron examined five samples (*P.J.*, 1900, May 26); none of them were of B.P. quality, and three of them were of less than one-tenth of it.

PROSECUTIONS. *London, Lambeth.* Deficient in dissolving power on white of egg to the extent of 60 % of the dissolving power required by the B.P. The defence was that "pepsin" was a name applied to various kinds of pepsin other than the B.P. article. The stipendiary said there was no suggestion of fraud, but that when "pepsin" was asked for the B.P. article must be supplied, and fined the defendant 5*s.* and costs (*P.J.*, 1904, Sept. 10; *B.F.J.*, 1904, 197).

London, North. The prosecution only asked for a nominal penalty as the deficiency in a number of samples was considered to be due to the manufacturers. Fine and costs 13*s.* 6*d.* (*P.J.*, 1908, Dec. 12).

WOOD CHARCOAL

Three of the sixteen Birmingham samples examined exceeded the B.P. limit of 7.5 % of ash, containing 10.0–12.0 % of ash; one of them had 4.0 % of ash insoluble in dilute HCl. Five samples had 2.7–4.2 %, and the other eight 5.2–7.0 %. Eight samples had 0.3–1.2 % of insoluble ash. Six samples absorbed 7.4–10.4 % of moisture in forty-eight hours after drying one hour at 120° C. (Brindle's test, *B.P. Conf.*, 1928, 330).

SACCHARIN, GLUSIDE

Methods for the examination of commercial saccharin have been given by Richmond and Hill (*J.S.C.I.*, 1918, 246T; 1919, 8T; also *Analyst*, 1918, **43**, 353; 1919, **44**, 99), and McKie (*J.S.C.I.*, 1921, 150T; also *Analyst*, 1921, **46**, 335). Lerrigo and Williams, also, have studied the methods for determination (*S.P.A.*, 1927, **52**, 377).

Schowalter has given a method for the separation of it from benzoic acid (*Analyst*, 1920, **45**, 266), and Richmond, Royce and Hill have discussed the examination of saccharin tablets (*S.P.A.*, 1918, **43**, 402). Methods for its detection and estimation in foods have been given by Klostermann and Scholta (*Analyst*, 1916, **41**, 309) and Bonis (*Analyst*, 1917, **42**, 303).

The following method was found in the writer's laboratory to be satisfactory for determining saccharin in cornflour, as in custard powder. Shake 20 gm. (or more) with 100 ml. cold water, filter next morning, and wash. Divide filtrate into two equal portions. (1) For blank. Add a little bromine water, allow to stand for a few hours, add 2 gm. of fusion mixture, boil off excess of bromine, and precipitate with BaCl_2 . The BaSO_4 represents any sulphates or sulphites present. (2) Evaporate to dryness on water bath, moisten with solution of Na_2CO_3 , and again evaporate. Add 2 gm. of fusion mixture and fuse for half an hour, flame gases being excluded. The fused mass is treated with water, acidified with HCl , filtered if necessary, and SO_4 determined. After subtraction of the blank, multiplication of the SO_4 by 1.91 will give saccharin.

PROSECUTIONS FOR SACCHARIN TABLETS. *Manchester.* The tablets contained only $\frac{1}{7}$ grain instead of the $\frac{1}{2}$ grain ordered. Fine £10 (*P.J.*, 1918, Nov. 23; *B.F.J.*, 1918, 135).

Batley. False trade description, and sale, of "half-grain" tablets which only contained $\frac{1}{3}$ grain. £10 fine for each of eight offences (*P.J.*, 1920, April 24).

PRECIPITATED SULPHUR, MILK OF SULPHUR

The B.P. of 1898, by giving "Milk of sulphur" as a synonym for "Precipitated sulphur," practically settled an old controversy. Sulphur, which has been dissolved by boiling with milk of lime, is reprecipitated on addition of acid. When hydrochloric acid is used, the dried precipitate rarely contains more than 0.4 % of ash, as the lime salts are in solution. When sulphuric acid is used, about three times as much product is obtained, the ash of which is about 50%, corresponding to about 60 % of calcium sulphate ($\text{CaSO}_4, 2\text{H}_2\text{O}$). Probably owing to deficient chemical knowledge, "Lac Sulphuris," prepared according to the London Pharmacopœia of 1721, might have been either practically pure sulphur or the calcarious article. The next edition, 1746, changed the name to "Sulphur Precipitatum," and ordered the calcarious article only. From 1788, Pharmacopœias only recognised the pure article. In a Birmingham prosecution in 1877, the defence argued that precipitated sulphur (pure) and milk of sulphur (calcarious) were two different articles, but the stipendiary convicted. A discussion of the subject followed a paper by A. Hill (*Analyst*, 1877, **1**, 57, 63).

The unsatisfactory position at the time is shown by the fact that about a quarter of the Birmingham samples of milk of sulphur were adulterated with sulphate of lime, and a similar proportion of those sold as precipitated sulphur; in one case the calcarious article was dispensed on a prescription. In later years the sale of the adulterated article was uncommon. Occasionally flowers of sulphur have been detected.

In 1905, there were prosecutions in various places for the presence of arsenic, and the B.P. of 1914 prescribed a limit of 5 parts per million.

Of the samples of "sulphur" bought in England and Wales during 1900-13, 5.6 % were reported adulterated, and 2.7 % of the samples bought as "sulphur and its preparations" during 1922-30.

PROSECUTIONS. *Birmingham.* Sulphate of lime 67 %. Nominal penalty of 1s. Notice of appeal was given, but not proceeded with (*Analyst*, 1877, 1, 57).

Chesterfield. Orange sulphuretted antimony. Fine £2 10s. (*Analyst*, 1897, 1, 118).

Birmingham. Sulphate of lime 57 %. Fine £2 (*F. & S.*, 1894, Aug. 25; *P.J.*, 1894, 54, 149).

London, Kensington. Sublimed or powdered sulphur 100 %. Fine 5s. (*B.F.J.*, 1900, 238).

Malmesbury. Arsenic $\frac{1}{8}$ grain per oz. (about 300 per million). The impurity was attributed to the hydrochloric acid used in making it. Fine 1s. (*Grocer*, 1905, July 8; *B.F.J.*, 1905, 145).

REDUCED IRON

The metallic iron content has been progressively increased by subsequent Pharmacopœias. In 1885 it was required to be at least 50 %, in 1898 at least 75 %, and in 1914 at least 80 %. The arsenic limit is 200 per million.

Williams and Anderson have described a modified copper sulphate method which gives an accurate determination of the metallic iron (*Analyst*, 1923, 48, 77). Dott found that ignition would not completely oxidise the iron, but that by first converting into nitrate, ignition produced Fe_2O_3 (*Y.B.P.*, 1924, 442).

PROSECUTIONS. *Marlborough.* Arsenic 1 grain per oz. The Secretary to the Pharmacopœia Committee stated that he did not think that proportion would be injurious to a patient. The Bench, hoping the publicity given would stop the practice, dismissed the case (*P.J.*, 1904, Oct. 8).

Malmesbury. Arsenic 7 grains per oz. The Public Analyst did not admit that arsenide of iron, if present, would be insoluble in the system. Fine 10s. (*P.J.*, 1904, Nov. 5).

OXIDE OF IRON

The 1885 B.P. contained " Peroxide of Iron," but it has not been in subsequent editions.

PROSECUTION. *Chippenham.* Calcium sulphate 54 %, moisture 16 %, silicious matter, alumina, etc., 10 %, and only 20 % iron peroxide. The question of standard was raised, as the article was not in the current Pharmacopœia, and the inspector pointed out that a purchaser was entitled to receive an article containing more than one-fifth of iron oxide. The defendant was unaware that it was incorrect, as he bought it with the business, and was ordered to pay costs only (*P.J.*, 1905, April 14).

SODIUM PHOSPHATE

The amount of arsenic allowed is now limited to 5 parts per million.

PROSECUTION. *Oxford.* Arsenic, calculated to arsenious oxide, 5 grains per lb. The Medical Officer of Health considered the proportion found was likely to be injurious to health. The magistrates fined the defendant 1s. only, as they believed the presence of arsenic was unknown to him, and that it entered in the process of manufacture (*P.J.*, 1900, Oct. 20).

MERCURY WITH CHALK, GREY POWDER

One-third of the B.P. preparation is mercury, but particles of mercury visible to the eye must not be present, and mercuric compounds must be absent. There is a possibility, that in an unremoved bottle, a larger proportion of mercury may be present at the bottom, so bottles should be shaken before dispensing (Dechan and Maben, *B.P. Conf.*, 1884, 556).

PROSECUTIONS FOR GREY POWDER. *Hull.* Phenazone 100 %. The mistake was made by an unqualified assistant. Fine £20 (*P.J.*, 1914, Nov. 19).

London, South-Western. Mercury deficient 64 %, and containing sugar of milk. Four powders were bought, mixed, and divided into three parts. It was argued that the division was incorrect, as each powder should have been divided, but the objection was overruled. The sugar of milk was stated to have been added as a safeguard to the child. Paid costs (*P.J.*, 1914, Mar. 14).

Lurgan. Mercurial compounds equivalent to 0.3 % of mercuric mercury. The Public Analyst considered the article had deteriorated through being kept too long in stock. Medical evidence was given that so small a proportion of the impurity would cause no harm whatever. Fine 6d. and £1 costs (*P.J.*, 1915, Oct. 16; *B.F.J.*, 1915, 198).

Exeter. Chiefly calomel, no grey powder. The Medical Officer

of Health said that calomel was more drastic. The case was dismissed on the technical objection that though six powders were bought, only one was analysed (*B.F.J.*, 1920, 17).

COMPOUND LIQUORICE POWDER

This powder, prepared according to the 1914 B.P., contained 2 % more sugar than the previous preparations. The table below gives the proportion of the constituents, with their composition and that of a powder made from them, compared with its calculated composition :—

COMPOSITION OF COMPOUND LIQUORICE POWDER

PERCENTAGES.	B.P. Proportions.	Ash.	Soluble in Water, 2 w/v.	Sand.	Total Sulphur.	Crude Fibre.
Senna leaves . . .	16	12.6	33.0	2.9	0.05	5.3
Liquorice root . . .	16	4.6	22.8	0.3	0.13	14.6
Fennel fruit . . .	8	9.5	23.0	0.5	0.16	14.8
Sublimed sulphur . .	8	0.1	—	—	99.9	—
Refined sugar . . .	52	—	100.0	—	—	—
— — —						
100						

Compound liquorice powder :

Calculated from constituents	3.5	60.7	0.5	8.04	4.4
Found by analysis . . .	4.8	58.6	0.7	7.98	4.8

The difference between the found and the calculated ash is due to the action of the sulphur on the ash constituents. The 2.1 % difference in soluble matter may be due to the plant constituents being less soluble in sugar solution than in water. Variations in the percentage of total ash are largely due to the proportion of sand (ash insoluble in HCl) ; in thirty-four Birmingham samples examined 1922–30, it varied 0.2–1.2 %. The following tabulation of them shows the effect of subtracting the sand :—

ASH IN COMPOUND LIQUORICE POWDER

PERCENTAGES . . .	2.9–	3.2–	3.7–	4.2–	4.7–5.4	Total.
PERCENTAGES OF SAMPLES :						
Total ash	0	0	29	44	27	100
Total ash, less sand.	3	20	65	12	0	100

After subtraction of the sand, two-thirds of the samples were 3.7–4.1 %. The amount soluble in water varied from 55.6–57.0 %, though 55 % of them only varied 63.0–65.8 %. The total sulphur, which includes the small amount of sulphate present in the organic powders, varied from 7.5–9.6 %, though 75 % of them were 7.9–8.4 %. The moisture varied from 2.9–5.2 %, though 83 % only varied 3.2–4.6 %. The amount of sulphate, determined directly on the powder, in ten samples was only 0.02–0.04 %.

During 1894–1930, 112 samples of the powder were examined, five of which were condemned ; four samples, prepared in war-time,

contained rice starch instead of sugar, and one sample 9.7 % of sulphur. Of the samples examined in England and Wales, 1903-13, 3.5 % were adulterated, and 2.3 % of those during 1920-30.

ANALYSIS. The amount soluble in water is determined by adding 100 ml. of water to 2 gm., shaking during the second day, and filtering on the third day, and determining the solids on 25 ml. of the filtrate. With insufficient shaking the value found may be decidedly low. The filtrate should have a green colour due to senna.

To determine sulphur add 20 ml. of HNO_3 to 1 gm., heat on water bath with periodical addition of KClO_3 . When the sulphur appears to be dissolved, transfer to a basin, add 20 ml. of HCl , and evaporate to dryness on a water bath, repeat evaporation after moistening with HCl . Boil with 15 ml. 3E.HCl and 100 ml. of water, filter and wash; make up volume of filtrate to about 200 ml. and precipitate with BaCl_2 . If the residue contain globules of sulphur, they must be picked out, weighed, and added to the amount of sulphur determined as sulphate. This method is more satisfactory than extraction with CS_2 .

Papers on the analysis of the powder have been given by Evans (*P.J.*, 1905, March 11), Parkes and Major (*S.P.A.*, 1914, **39**, 160), and Scott-Smith and Evans (*S.P.A.*, 1911, **36**, 198); the latter deals with the microscopical examination of it, and gives plates. Ground olive stones and almond shells have been detected by them.

PROSECUTIONS. *Saddleworth.* Destitute of sulphur. For the defence fraud was denied, as the powder without sulphur was 50 % dearer than the B.P., and it was stated people preferred it without sulphur. Fine 5s. (*F. & S.*, 1894, June 2).

Bradford. Sulphur 23 %, liquorice 26 %, sugar 50 %. Paid costs (*B.F.J.*, 1899, 345).

Wimbledon. Sulphur 5.2 %, moisture 3.8 %, senna, liquorice root and sugar 91 %; deficiency of sulphur 3.1 %. The certificate was objected to as it did not give the exact proportions of everything found. The magistrates, following the Appeal Case, *Fortune v. Hanson* (1896), did not think the certificate gave all the information they were entitled to, and dismissed the case (*P.J.*, 1903, May 23).

Preston. Ground olive stones 6 %. The defence was that the ground liquorice used had been adulterated in Marseilles. Fine 5s. (*P.J.*, 1904, Dec. 17).

Sheffield. Ground almond shell 5 %. Paid costs (*P.J.*, 1911, Feb. 27).

Birmingham. Devoid of sugar; rice starch had been used instead. Fine £1 (1918 Report).

COMPOUND POWDER OF CINNAMON

According to the editions of the B.P. from 1867-1914, this powder has been prepared from equal weights of ground cinnamon,

ginger and cardamoms. A sample bought at Crewe (*P.J.*, 1912, May 4; *B.F.J.*, 1912, 95) was adulterated with 45 % of chalk. Objection was taken to the summons describing it as an article of food, when it was a drug. It was said to have been prepared by "an older and more expensive formula." Possibly aromatic powder of chalk was intended, but that only contained 25 % of chalk, and is much cheaper than the drug asked for. The case was dismissed.

COMPOUND RHUBARB POWDER, GREGORY'S POWDER

The difference in composition of the 1914 B.P. powder from that suggested for the 1932 B.P. is shown below :—

1914.	Rhubarb, 22 %,	Ginger, 12 %,	Light magnesia . . .	66 %
			Light carbonate of	
1932.	„ 25 %,	„ 10 %	magnesia . . .	32.5 %
			Heavy carbonate of	
			magnesia . . .	32.5 %

Most of the prosecutions of the past have been due either to the deterioration of the light magnesia by careless keeping, or by the substitution of carbonate for oxide. As samples of the 1914 powder will probably be sold for some time, methods for its analysis are given subsequently.

The following table relating to the two powders and their constituents is based on data previously given, further particulars may be found in a paper by Liverseege, Bagnall and Lerrigo, given to the British Pharmaceutical Conference (*B.P. Conf.*, 1926, 465) :—

COMPOSITION OF GREGORY'S POWDER AND CONSTITUENTS

PERCENTAGES.	Rhubarb.	Ginger.	Light Magnesia.	Carbonates of Magnesia.	Gregory's Powder, B.P. 1914.	B.P. 1932.
Ash	10	5	98	43	67.5	31.0
Loss in water oven .	7	10	1	2	3.4	4.0
Carbon dioxide . .	0	0	1	35	0.7	22.7
Organic matter and combined water .	83	85	0	20	28.4	42.3
Total	100	100	100	100	100.0	100.0
Water extract . .	40	16	1.2	1.2	11.9	12.8
Insoluble in acetic acid .	52	78	—	—	20.8	20.8

CARBON DIOXIDE IN GREGORY'S POWDER, 1902-25

(Sixty-seven samples)

Carbon dioxide, % .	0.6-2.8	3.0-6.2	12.0-14.1	20.3-28.0	Total.
Percentage of samples .	66	16	9	9	100

Although, according to the previous table, 0.7 % of carbon dioxide should be present, that figure is not a reasonable limit, except for samples very recently prepared. On exposure to air the magnesia gradually absorbs moisture and carbon dioxide, forming light carbonate of magnesia and possibly hydrate. The above

figures show that with reasonable care in keeping, the carbon dioxide need not amount to 3 %. On this basis there are three groups of samples :—(A) Thirty-eight samples, containing less than 3 % of carbon dioxide, which were passed as genuine. (B) Fourteen samples, which had probably been correctly made, but had deteriorated, owing to unsatisfactory conditions of keeping. (C) Five samples from three vendors, in which carbonate had been substituted for magnesia. The range of composition of the three classes is given below :—

THREE CLASSES OF GREGORY'S POWDER

PERCENTAGES.	CLASS A.	CLASS B.	CLASS C.
Carbon dioxide	0.6- 2.8	3.0-14.1	20.3-28.0
Ash	63.6-72.5	44.2-67.4	30.6-45.9
Loss in water oven	1.8- 3.6	2.8- 7.7	3.0- 5.1

With the increase in the proportion of carbon dioxide, the percentage of ash falls, and usually the percentage of loss in the water oven increases. While the theoretical percentage of moisture in Gregory's powder (1914), as shown above, was 3.4 %, 60 % of the samples passed as genuine lost less than 3.0 % on drying. Probably part of the moisture was fixed by the magnesia, though one has heard of *dried* rhubarb and ginger being incorrectly used.

ASH OF NORMAL GREGORY'S POWDER (Thirty-eight samples)

Ash, normal samples, % .	63.6-	65-	66-	67-	68-	69-72.5	Total.
Percentage of samples .	11	18	11	26	16	18	100

Owing to the absorption of moisture and carbon dioxide many of the samples are below the theoretical 67.5 % of ash in freshly prepared powder.

For the other analytical constants, the proportion of carbon dioxide makes little difference, and all samples are taken for the calculation of the percentage ranges :—

ANALYSES OF GREGORY'S POWDER

ORGANIC MATTER, % .	22-	23-	24-	25-	26-	27-	28-	29-32	Total.
Percentage of samples	2	6	4	14	19	33	14	8	100
ACETIC ACID INSOLUBLE, %	17.2-		19-	20-	21-		22-23.8		Total.
Percentage of samples	16		18	34	16		16		100
WATER EXTRACT, % .	10.8-	11.0-		11.5-	12-	12.5-	13-13.5		Total.
Percentage of samples	6	16		22	25	12	19		100
SPIRIT (S.V.M.) EXTRACT, %	4.1	5-	6-	7-		8-9.1			Total.
Percentage of samples	4	31	31	23		11			100

The above tables indicate that the usual ranges of the 1914 powder were—organic matter 25-28.9 %, insoluble in acetic acid 19-21.9 %, water extract 10.8-12.9 %, and methylated spirit extract 5-7.9 %.

The percentage of adulteration in England and Wales was, in 1898-9, 27.8 %; in 1905-13, 12.3; and in 1920-30, 12.0.

ANALYSIS. The sp. gr. is determined by putting 20 ml. into a weighed cylinder about 18 mm. in diameter, only shaking to level the surface, and weighing again. If prepared with *light* magnesia, it should be about 0.17–0.20. The moisture and ash are determined on 1 gm., wetting with spirit to avoid mechanical loss. The carbon dioxide may be determined gravimetrically or by Hepburn's method. The matter, chiefly organic, insoluble in acetic acid is determined by treating 1 gm. with 100 ml. E/2 acetic acid overnight, filtering through tared filter paper, washing once with water, and drying four hours or more.

The methylated spirit extract is determined by adding 150 ml. to 1.5 gm. of the powder in a corked conical flask, shaking thoroughly during the second day, allowing to stand all night, then decanting without shaking, filtering if necessary, evaporating 100 ml. to dryness, and drying in the water oven for three hours. The water extract can usually be determined in the same way, but some samples of magnesia, owing to their physical condition, reprecipitate the dissolved organic matter, giving low results. The method is then modified as follows:—Instead of allowing to stand, shake continuously for one minute, throw the whole on to a dry 18½ cm. filter, No. 595. Return the first few ml., evaporate 100 ml. of the clear filtrate as before. It should be noted that the amount of water-soluble extract depends on two factors—the amount of soluble matter in the rhubarb and ginger, and also on the quantity of liquid evaporated. In the above conditions, the amount of magnesia or magnesium carbonate soluble in 100 ml. of liquid will increase the rhubarb and ginger water-soluble extract by 1.2 %.

Calculation. As previously explained under “magnesia,” multiplication of the carbon dioxide by 0.55 will give the corresponding amount of water in light carbonate. Subtraction from 100 of the sum of ash, loss in water oven, carbon dioxide and corresponding water, will give the organic matter. Multiplication of the organic matter by 1.19 will give the total amount of rhubarb and ginger present. An alternative method is to multiply the sum of the acetic acid insoluble matter and the water extract by 1.05.

The proportion of light carbonate may be obtained in two ways : (A) by multiplying the carbon dioxide by 2.8, or (B) by the following equation, where “a” = percentage of ash, and “b” the total percentage of rhubarb and ginger present :—

$$\text{Carbonate, \%} = \frac{(67.5 - a)(100 - b)}{35.7}.$$

Papers on the analysis of Gregory's powder and its deterioration have been given by Paul and Cownley (*P.J.*, 1898, Oct. 8), J. C. Umney (*C. & D.*, 1898, Sept. 24), Harvey (*C. & D.*, 1900, Sept.), and Elsdon and Hawley (*B.P. Conf.*, 1915, 403).

Examples. The following are selected from the paper previously mentioned (*B.P. Conf.*, 1926, 465). I. was made in the laboratory. II. was a freshly made wholesale sample. III. was twenty-four years old. IV. had deteriorated on keeping. V. was made with light carbonate instead of magnesia. VI. was made with 12 % of rhubarb, and 22 % of ginger, instead of the proper quantities :—

ANALYSES OF GREGORY'S POWDER

PERCENTAGES . . .	I.	II.	III.	IV.	V.	VI.
ANALYTICAL RESULTS.						
Ash	67.6	68.7	66.2	47.6	32.3	66.7
Carbon dioxide	0.6	0.9	2.1	12.1	23.3	0.3
Corresponding water ($\text{CO}_2 \times 0.55$)	0.3	0.5	1.2	6.7	12.8	0.2
Loss in water oven	2.6	2.1	1.7	4.5	3.9	3.0
Organic matter (by difference).	28.9	27.8	28.8	29.1	27.7	29.8
Total	100.0	100.0	100.0	100.0	100.0	100.0
Acetic acid insoluble	20.7	20.3	20.2	20.2	20.2	24.3
Water extract	11.2	12.2	12.6	11.9	11.5	9.4
TOTAL RHUBARB AND GINGER.						
1.05 (acetic acid insoluble + water extract)	33.5	34.1	34.3	33.7	33.3	35.4
Organic matter $\times 1.19$	34.6	33.1	34.3	34.6	33.1	35.5
CARBONATE OF MAGNESIA.						
By equation (A)	2	2	6	34	65	1
By equation (B)	0	0	2	37	66	1

The above figures show that the two independent methods of calculating the total rhubarb and ginger give similar results, and that the two calculations of carbonate show little difference.

Sample III., after keeping for twenty-four years in a corked bottle, only contained 2.1 % of carbon dioxide ; the loss in the water oven had *decreased* from 3.6 %. A sample of Gregory's powder reported on by Elsdon and Hawley (*opus cit.*) had been exposed to the air for over three months (July–November) outside the laboratory in Birmingham. It gained 12.3 % in weight, and the carbon dioxide increased from 2.2 % to 3.5 %. This was, of course, a severe test. The deterioration of this drug is not so rapid as has been sometimes suggested.

PROSECUTIONS FOR GREGORY'S POWDER. *Lambeth.*

Official carbonate of magnesia 32.41 %, light magnesia 34.43 %. The use of carbonate was denied by the defence, and its presence attributed to change on exposure, which was stated not to affect its medicinal value. Fine £2 (*C. & D.*, 1898, Aug. 27 ; *F. & S.*, 1898, Aug. 27).

Watford. Magnesium carbonate instead of oxide. For the defence it was pleaded that carbonate was used in other countries, and that, while the B.P. was the standard for a prescription for "Pulv. Rhei Co.," it was not for "Gregory's powder." The defendant

was warned to follow the B.P. directions in the future, and the case was dismissed (*C. & D.*, 1900, Sept. 15 ; *B.F.J.*, 1900, 297).

Kensington. Calcium carbonate 65.6 %. The owner of the drug store, where it was sold, said a mistake had been made. Fine £5 (*B.F.J.*, 1901, 397).

London, Marylebone. Magnesia, 29.6 %. The defendant said some of his customers preferred half the usual quantity of magnesia. Fine 5s. and costs (*P.J.*, 1908, Dec. 26).

London, Old Street. Carbonate of soda 15 %, basic carbonate of magnesia 41 %, and no magnesium oxide. Fine £4 (*B.F.J.*, 1909, 32).

Birmingham. Magnesium carbonate 4 parts, rhubarb and ginger 1 part. Fine 10s. A previous purchase of "compound rhubarb powder" contained 60 % of magnesium carbonate. The unqualified vendor apparently did not know the two names represented the same drug (1910 Report).

London, Clerkenwell. Rhubarb root in powder 100 %. The substitution was said to be a mistake between two similarly labelled bottles. Paid costs (*P.J.*, 1911, April 1 ; *B.F.J.*, 1911, 76).

Hungerford. Light magnesium carbonate 66 %. The article was stated to be practically identical with "Improved Gregory's powder" of the British Pharmaceutical Codex. Case dismissed (*P.J.*, 1924, March 12).

PROSECUTION FOR COMPOUND RHUBARB POWDER.

West Ham. "Not genuine compound rhubarb powder according to the formula of the British Pharmacopœia—it contained only 30 % of magnesia, whereas 66.6 % is prescribed by the British Pharmacopœia." The inspector did not present a prescription, nor specify "B.P." The magistrate was of opinion that the analyst's certificate did not prove the case. He did not say it was not genuine, but it was not in accordance with the British Pharmacopœia, and that there was no Act of Parliament making that the standard. The case was dismissed and the defendant allowed 10s. costs (*C. & D.*, 1900, Jan. ; *B.F.J.*, 1900, 27, 16).

CHAPTER XXXIV

ESSENTIAL OILS. MEDICINAL SPIRITS. TINCTURES

Essential oils :—Eucalyptus. Juniper. Turpentine. Camphor. Spirits :—Camphor. Chloroform. Nitrous ether, sweet nitre. Aromatic spirit of ammonia, sal volatile. Essence of peppermint. Compound tincture of benzoin. Tincture of myrrh. Tincture of opium, laudanum. Paregoric. Tincture of rhubarb. Tincture, and ammoniated tincture, of quinine. Tincture, and strong tincture, of iodine. Iodine paint.

ESSENTIAL OILS

THERE have been very few prosecutions for adulterated essential oils, and the number of samples taken has not been large. During 1905-7, 149 samples were taken in England and Wales, and 8·7 % of them were adulterated.

The Society of Public Analysts appointed a Sub-committee to consider the analysis of essential oils, and eight Reports have been published in the *Analyst*. Estimation of cineole in eucalyptus and other oils (1927, **52**, 276 ; 1931, **56**, 738). Physical constants (1927, **52**, 530 ; 1929, **54**, 335). Acetylisable constituents (1928, **53**, 214). Phenols (1928, **53**, 215). Citral in lemon oil (1930, **55**, 109). Solubilities (1930, **55**, 386). For carvone, see Bennett and Cocking (*S.P.A.*, 1931, **56**, 79).

OIL OF EUCALYPTUS

All of the eighteen Birmingham samples of the oil examined 1922-30 were within the B.P. limits. The sp. gr. of fifteen ranged 0·918-0·925, the extremes being 0·915 and 0·927. All the rotations were positive, and thirteen of them were 0·5° to 1·5° in 100 mm. tube, the highest was 4·0°. The cineole of some of the samples was determined by the B.P. method, and of others by the Committee's method (*opus cit.*), the ranges were similar, 65-85 v/v. The B.-R. 20° readings of fifteen of them were 52·4 (R.I. 1·4607-20), the extremes being 50 and 55 (R.I. 1·4593 and 1·4628).

In England and Wales two of the 238 samples examined 1908-13 were adulterated, and two of the 468 during 1920-30.

PROSECUTIONS. *Clerkenwell.* Oil not conforming to the requirements of the B.P. for oil of eucalyptus, 100 %. Samples from three vendors had sp. gr. 0·865-0·890, and negative rotations 29-65°. They were deficient in cineole and contained phellandrene. It was stated that the oils were from *Eucalyptus amygdalina*, which

was permitted by the 1885 B.P. Each vendor was fined 5s. (*C. & D.*, 1899, Jan. 7 ; *B.F.J.*, 1899, 24).

Lambeth. Oil not in accordance with the requirements of the B.P., 100 %. Fine £2 (*P.J.*, 1909, Dec. 4 ; *B.F.J.*, 1910, 10).

OIL OF JUNIPER

The B.P. article is distilled from ripe juniper berries, not unripe, as the 1885 B.P. stated. There is also a juniper wood oil, about half the commercial value of the genuine oil, and the tests of the 1898 B.P. were insufficient to distinguish the two oils. The rotation of the wood oil is greater than that of the genuine, say -17° to -36° in 100 mm. tube. According to Parry, some samples of this oil are largely turpentine. At the British Pharmaceutical Conference, 1907, papers on the oil were given by Umney and Bennett (p. 273) and Bird (p. 276).

PROSECUTION. *North London.* Oil of turpentine 70 %. Fine £1 (*P.J.*, 1904, Aug. 5).

Clerkenwell. Oil of turpentine 68 %. The report of the Government analysts was that "We find the sample complies with the requirements of the B.P." For the defence it was argued that this indicated that the article was genuine. The Public Analyst gave evidence that the B.P. tests were antiquated and that a mixture of oil of turpentine and juniper would pass them. The question was referred to a Committee jointly appointed by the Society of Public Analysts and the Pharmaceutical Society. The Committee decided that the 1898 B.P. characters and test were an insufficient guide for judgment of purity (*P.J.*, 1907, May 18, July 13, Aug. 17 ; *B.F.J.*, 1907, 104, 125).

TURPENTINE

"Turpentine," strictly speaking, is the oleo-resin which exudes into holes cut in the trunks of pines, and which, by distillation with water, is separated into rosin and oil, or spirit, of turpentine ; but the term is commonly applied to the oil itself. The defence in prosecutions is often that the article sold was not the drug but the commercial quality, and *therefore* it was mixed. Although the B.P. article is described as "rectified oil of turpentine," it differs little from the genuine commercial quality, and the sale of "white spirit," or other petroleum substitute, as "turpentine" is an offence under the Merchandise Marks Act. When a sample is bought under the Sale of Food and Drugs Act, even from a pharmacy, some indication must be given to the vendor that it is required for medicinal use, and not for paint or polish.

The analysis of fifty-seven samples supplied to Birmingham Corporation Departments as "turpentine," and which were passed

as genuine, gave the following results :—The sp. gr. of forty-five of them was 0.867–0.870, and the extremes were 0.864 and 0.873. The R.I. 25° C. of forty-four were 1.4672–1.4685, the extremes being 1.4659 and 1.4697. The rotations in 100 mm. tube varied from – 2.2° to + 5.1°. Of twenty-two samples given two treatments by Armstrong's method, nineteen gave residues 4–8 v/v., the extremes being 3 v/v and 10 v/v. The R.I. of sixteen of these residues were 1.4730–1.4772. As a contrast to these a sample of "white spirit" had a sp. gr. 0.792, and R.I. of 1.4375. This refraction was unaltered by Armstrong's treatment, and the residue measured 98 v/v. A sample of resin spirit had a sp. gr. 0.872, R.I. 1.4739, but yielded 43 v/v of residue by Armstrong's method, with R.I. 1.4524. There was a marked difference in the iodine values, the white spirit was 8, the resin spirit 141, while genuine turpentine is about 300.

An experience with an adulterated turpentine may be given as an illustration of the important principle that any evidences of adulteration should be kept for some time for reference. I condemned a sample of turpentine as containing "at least 10 % of a petroleum product." The contractor was brought to see me. He protested that his article was pure, and said another analyst should examine it. I gave him his "turpentine" to smell, and then another bottle containing the residue from Armstrong's method. He said, "That is very different; it is petroleum." My reply was, "It came out of your article." There was no further talk of another analysis.

There have been numerous papers on turpentine and its substitutes, among which may be mentioned those by McGill (*J.S.C.I.*, 1907, 847), Coste (*S.P.A.*, 1908, **33**, 219; 1910, **35**, 112), Coste and Nash (*S.P.A.*, 1911, **36**, 207), Richardson and Bowen (*J.S.C.I.*, 1908, 613), Richardson and Whitaker (*J.S.C.I.*, 1911, 115). For the sulphuric acid treatment, see Armstrong (*J.S.C.I.*, 1882, 479), Coste (*S.P.A.*, 1909, **34**, 148), and Morrell (*J.S.C.I.*, 1910, 241), and for the halogen absorption value, Worstall (*J.S.C.I.*, 1904, 302), Harvey (*J.S.C.I.*, 1904, 413), and Taylor (*S.P.A.*, 1919, **44**, 401).

Of the samples of turpentine examined in England and Wales, 1909–11, 8 % were reported adulterated.

ANALYSIS. Determination of *petroleum spirit* by modified Armstrong's method. Add 100 ml. of turpentine to a cooled mixture of 20 ml. of B.P. sulphuric acid and 10 ml. of water, agitate carefully, cool as temperature rises, and continue agitation as long as heat is developed. If an emulsion results, distil with steam, change receiver for each 100 ml. distilled, separate and measure the oil. To this distilled oil, or to the separated oil if there has been no emulsification, add about half its volume of a cooled mixture of 4 volumes of sulphuric acid with 1 of water, shake, cool, remove

the acid liquor by a separator, distil oil with steam as before, measure and determine refraction.

Beading test. Nash (*S.P.A.*, 1909, **34**, 151) has shown that on shaking turpentine the froth formed breaks immediately, and that the amount of frothing is proportional to the amount of petroleum spirit present.

By simple distillation, using a straight uncooled tube as a condenser, turpentine will yield 92 v/v or more of distillate between 152° and 165° C. Substitutes will begin to distil at a lower temperature and yield a large proportion over 165°.

PROSECUTIONS FOR SPIRIT OF TURPENTINE. *Bradford.* Gasoline 42 %. The article had been sold in penny bottles. Fine £5 (*P.J.*, 1909, May 1; *B.F.J.*, 1909, 93).

Bradford. Petroleum, 15 %. Fine 10s. (*P.J.*, 1910, Nov. 12).

PROSECUTIONS FOR TURPENTINE. *Stockport.* Lighter fractions of petroleum 10 %. A quart had been purchased and the defendant said that when required for medicinal use only ounces were bought, also that the drug was "oil of turpentine," and not "turpentine." The magistrates dismissed the case as the article had not been sold as a drug (*P.J.*, 1907, Sept. 28; *B.F.J.*, 1907, 180).

London, Old Street. Entirely petroleum derivatives. Evidence was given by a previous customer that she had rubbed some on her chest for a cold, and that it had caused agony and blisters. The defendant, who kept an oilshop, was fined £20 (*P.J.* and *Grocer*, 1925, May 7; *Analyst*, 1925, **50**, 185; *B.F.J.*, 1925, 50).

Birmingham. Mineral oil at least 30 %. Fine 10s. (1931 Report).

PROSECUTION FOR "FINEST HOUSEHOLD TURPS." *Salford.* Applying a false trade description to an article which contained 70 % of paraffin of the nature of "white spirit." The manufacturers were fined £7 10s., and also £7 10s. for aiding, abetting, counselling, or procuring the trading firm to commit the offence (*Analyst*, 1931, **56**, 530; *B.F.J.*, 1931, 79).

CAMPHOR

The specific rotation of eleven Birmingham samples varied from 40.0–41.5°, the average being 41.1°. Synthetic camphor differs from the natural in being practically optically inactive. The non-volatile residue of these samples varied from 0.0 % to 0.12 %.

Solutions of camphor in 21 w/v solutions in olive, sesame, cottonseed, arachis and colza oils indicated that the specific rotation of camphor in these conditions was about 54°, correction being made for the rotation of the oil itself. See also Schroff (*Q.J.P.*, 1929, 464).

Two samples of camphor absorbed 1.3 % and 2.1 % of iodine

respectively. With specially refined camphor Salamon (*S.P.A.*, 1923, **48**, 536) found that the iodine value did not exceed 0.1 %.

For pharmaceutical preparations, Dowzard has given a method of determination of natural camphor (*Analyst*, 1914, **39**, 353), and Bougalt and Leroy for synthetic camphor (*Analyst*, 1928, **53**, 546). On the importance of synthetic camphor, see "Pharmaceuticus" (*P.J.*, 1930, May 31).

SPIRIT OF CAMPHOR

The B.P. preparation is a 10 w/v solution of camphor in 90 v/v alcohol. The specific rotation depends somewhat on the concentration. Dissolved in alcohol, sp. gr. 0.8388, 14 w/v gave specific rotation of 40.4° ; a 9 w/v solution gave 39.9° . Taking 40° , each degree of rotation in a 200 mm. tube represents 1.25 w/v of camphor, in the absence of synthetic camphor. With weaker spirit the specific rotation is less, 10 % of water reducing it to 38.5° , in which case the factor would be 1.30. Alcohol being more volatile than camphor, on keeping the proportion of the latter tends to increase. Kept in a corked bottle for seven years, 9.8 w/v increased to 10.0 w/v.

By the subtraction from the sp. gr. of 0.0012 for each 1 w/v of camphor present, an approximation is made to the sp. gr. of the alcohol present in the spirit of camphor. The spirit may also be tested by adding water to 10 ml. of it in a graduated cylinder till there is slight permanent precipitation; the total volume should be about 18 ml. Its sp. gr. should be about 0.846 (see Liverseege, *C. & D.*, 1899, Jan. 28).

Experiments on the rotation of camphor in solutions have been given by Partheil and Van Haaren (*J.S.C.I.*, 1900, 684) and Schroff (*Q.J.P.*, 1929, 464). Methods for the analysis of spirit of camphor have been given by Susser (*Q.J.P.*, 1928, 596), Meyer (*Q.J.P.*, 1929, 417), and Milner (*Q.J.P.*, 1930, 338).

PROSECUTION FOR SPIRIT OF CAMPHOR. *Cannock.* Camphor 23 % deficient, as compared with the B.P. Costs 1 guinea (*P.J.*, 1903, June 20).

SPIRIT OF CHLOROFORM

The B.P. requires this preparation to be a 5 v/v solution of chloroform in 90 v/v alcohol, and there appears to be no difficulty in making it, though care is necessary in storing it.

PROSECUTION. *Taunton.* Deficiency 61.4 %. The defendant said it was a physical impossibility to make the article in strict accordance with the B.P., and for it to contain the percentage of chloroform given, also that it had been kept some time. It was argued that there was no standard for the article sold over the counter. Case dismissed (*P.J.*, 1923, Jan. 20).

SPIRIT OF NITROUS ETHER, SWEET NITRE

A translation of the London Pharmacopœia of 1746 gave "Sp. Nitri Dulcis" as a synonym, distinguishing "sweet spirit of nitre" from "spirit of nitre," which was nitric acid. The former English term was first given in the 1898 B.P., and settled the dispute as to whether "sweet nitre" was the preparation of the London or of the British Pharmacopœia. The London preparation known as "Sp. Gr. 0.850" has been very popular and is still sold, though it often contains very little nitrous ether. The price is a few pence per lb. less than the B.P. spirit. There is also a still cheaper preparation, "Sp. Gr. 0.900." The two latter articles must not be sold as "sweet nitre" as their composition is different from the B.P. spirit, and at the present time they have a very small sale.

The medicinal action is chiefly due to ethyl nitrite, but ethyl nitrate and aldehyde have some action. Paraldehyde, nitrous, nitric and acetic acids are also present on keeping the spirit (Marshall and Gilchrist, *Brit. Med. Jour.*, 1915, July 24; *P.J.*, also *C. & D.*, 1915, July 31; *Y.B.P.*, 1916, 303).

Before 1885, when A. H. Allen published his method (*P.J.*, Feb. 21), the determination of nitrous ether in this preparation was unsatisfactory. The amount of nitric oxide gas yielded by a volume of the spirit when treated with iodide of potassium and acid was used by him as a measure of the amount of nitrous ether present. The subsequent Pharmacopœias adopted the method, and fixed the upper limit as 7 volumes of gas from 1 volume of the spirit. In 1914 the lower limit was decreased from 5 volumes to 4 volumes (= 1.25 w/v ethyl nitrite).

It is recommended that the limits for the 1932 B.P. shall be 1.25–2.5 w/v ethyl nitrite, corresponding to a yield of 4 volumes and 8 volumes, respectively.

The following is a tabulation of samples bought retail in Birmingham :—

NITRIC OXIDE FROM SPIRIT OF NITROUS ETHER
(seventy-three samples)

No. ml. of nitric oxide yielded by 1 ml. of spirit	0-	3-	4-	5-	6-	7.1-12	Total.
Percentage of samples :							
1885-90	56	12	8	12	8	4	100
1892-4	12	4	25	29	17	13	100
1895-9	4	13	8	25	29	21	100

The unsatisfactory nature of the first period is shown by more than two-thirds of the samples yielding less than 4 volumes of gas, while others were much too strong. Most of the inferior samples had been bought as "sweet nitre." In the two later periods the percentage of samples below 4 volumes had fallen to about 16 %,

and in the last period only 8 % more were between 4–5 volumes ; these would have passed the 1914 standard, but not the 1898 one. From 1892–7, spirit of nitrous ether was asked for.

The 1898 B.P. increased the alcoholic strength of the constituent spirit from 88·7 v/v to 90 v/v, and consequently the sp. gr. range of the preparation was altered from 0·840–0·845 to 0·838–0·842.

SP. GR. OF SPIRIT OF NITROUS ETHER

Sp. gr.	0·824–	0·838–	0·840–	0·845–	0·850–	0·860–	0·896–	0·947	Total.
Percentage of samples :									
1885–90	0	0	23	41	23		13		100
1892–4	13	4	42	33	8		0		100
1895–9	13	8	46	29	4		0		110

In the first period 13 % of the samples had been badly watered, and the proportion with sp. gr. 0·850–0·860 was much higher than in the other periods ; there was also a decreasing proportion of the 0·845– samples.

During the years 1894–1905, 27 % of the samples of sweet spirit of nitre examined in England and Wales were adulterated, and 23 % of those during 1920–30. In the intermediate period 1906–13, 26 % of the samples of spirit of nitrous ether were adulterated.

DETERIORATION ON KEEPING. The instability of this preparation is recognised by the B.P., as is shown by the allowance for a fall of strength and by the warning that it should be kept in small well-closed bottles in a cool dark place. While admitting this, it must be pointed out that the instability of the article has been grossly exaggerated in many statements made in the defence of prosecutions for the sale of defective samples. Some of these suggest that the retail sale of an article of satisfactory strength is impossible.

When freshly prepared, the spirit is free from aldehyde ; but on keeping, that substance is produced, and also acetic and other acids, all of which cause a diminution in the proportion of ethyl nitrite present (Marshall and Gilchrist, *opus cit.*).

The rate of change depends very much on the conditions, important factors being : (1) The relation of the liquid to the air space above it. (2) The frequency with which the bottle is opened. (3) Temperature. (4) Exposure to light.

Harvey (*C. & D.*, 1901, May 25) shook 1 oz. of the spirit in an 80-oz. bottle, and in five minutes half of its ethyl nitrite had been lost. More or less of the ethyl nitrite dissolved in the spirit vaporises into the space above, with a weakening of the spirit. If liquid be poured from such a bottle, the ethyl nitrite vapour is also poured out, and when the bottle is again closed, more ethyl nitrite is volatilised to take its place. Bottles should therefore be as full, and opened as rarely, as is possible. In other words, the size of the bottle should be in relation to the rate of sale.

Obviously a defective stopper will increase the rate. As an extreme case, Hodgson and Bailey (*P.J.*, 1914, Jan. 10) left the stopper out of a bottle, but four days were required to lose the difference between the B.P. maximum and minimum (1.1 %) of ethyl nitrite.

If bottles are unopened, loss is very slow. Southall's Report for 1902 mentions a sample, kept in a corked bottle coated with paraffin, which after five years yielded 4.8 volumes. In a later Report (1909), an experiment is described in which 1 oz. was poured out each week for thirteen weeks, and the spirit fell from 7.1 to 5.9 volumes. The bottles were exposed to some sunlight, and green or amber glass made little difference. In another experiment by Hodgson and Bailey (*opus cit.*), a sample took ten months to lose 1.1 % of ethyl nitrite; the stopper had been removed eighteen times for sampling. A still more striking test was made by Pittuck and Merson (*C. & D.*, 1900, Feb.) by taking ninety samples over a period of eight weeks. The spirit was kept in the light, but not in the sun, and fell from 6.6 volumes to 4.2 in that period.

A cool place for keeping is obviously desirable, as the solubility of the ethyl nitrite decreases with rise of temperature. According to Southall's Report for 1909, 4 fl. oz. in an open measure lost 4 % in half an hour at 11° C., but 1 fl. oz. lost 42 % in the same conditions at 18°.

Sunlight promotes decomposition. Shaw (*P.J.*, 1903, Aug. 8) half filled four ounce bottles and examined after seven days. The spirit in the one kept quite dark fell from 6.5 to 5.8 volumes, and one in full summer light to 4.9 volumes. Amber glass appears to give as great a protection as darkness. Harvey (*opus cit.*) kept samples in sunlight out of doors for seven days. The sample in colourless glass fell from 7.1 volumes to nothing, while one in amber glass lost only 0.5 % of strength. The sample in deep green lost 82 %, and one in deep blue 80 %. Another sample in amber glass kept four and a half months lost 12 %.

There have been many prosecutions for the addition of water to the spirit; besides the reduction of strength by dilution, decomposition is promoted by water. The effect of small quantities is not serious; an undiluted sample fell from 6.5 to 6.4 volumes in ten months; one with 5 % added water fell to 6.1 volumes, correction being made for the dilution (Southall's Report, 1912). Any statement that a sample has rapidly decomposed owing to a wet bottle is therefore not justified. The reduction in alcoholic strength on keeping is slight. Pittuck and Merson (*opus cit.*) record an alteration in sp. gr. from 0.8375 to 0.8390 in ten weeks, the bottle having been sampled 112 times.

There have been few figures published dealing with the alteration of acidity by keeping. A sample examined by Harvey (*opus cit.*)

had 5.5 N. v/v, which was doubled in eight months when kept in a bottle one-fifth full in the dark. This was a contrast to a sample which was kept in amber glass for four and a half months and only altered from 0.4 to 0.5 N. v/v. Burford (*C. & D.*, 1901, July 20) kept a sample in a bottle nearly full for a year exposed to light, when the acidity *fell* from 8.1 to 5.3 N. v/v; a parallel sample in a bottle half full gave an *increase* of acidity to 15.6 N. v/v.

It has been stated previously that about one-quarter of the samples examined were adulterated, but all were not due to deterioration; some had been watered, and inferior substitutes sold in other cases. Also, samples have been obtained from grocers, and from small shops, where the vendor may have been ignorant of the proper conditions for keeping the spirit, and where the sale has been very slow. In one prosecution, given below, the spirit had been in stock eight years.

SAMPLING. Correct sampling of sweet nitre requires more care than any other drug, and some suggestions are here given. Take a dry corked bottle of the right size, say 4 oz., or let the vendor supply the article in a bottle. Do not shake the sample, and divide it as quickly as possible, noting the time taken, between three well-stoppered bottles of $1\frac{1}{2}$ oz. capacity, replacing the cork each time. Retain the original bottle and label. Tie each bottle over with parchment paper, put each in an opaque envelope and seal securely. Both the bottles and the wrapper should be numbered. Sealing a corked bottle is unsatisfactory, as the spirit may dissolve the sealing-wax. One sample should be taken, or sent, to the analyst forthwith, and the third sample kept in a cold place. If possible, the analyst should see the inspector divide a sample of known composition, and determine the percentage of loss during the process. Evidence on this point would be invaluable, if the method of division be challenged, as has often been the case.

In this connection, the report of a prosecution in 1899, at Marylebone, given below (p. 493), should be read.

ANALYSIS. Owing to the decomposition caused by water the acidity is best determined by N/10 alcoholic KOH. Some indication of the acidity of a sample is given by the amount of gas liberated by KI, *before* the addition of H_2SO_4 ; 5 ml. is a convenient quantity of the spirit to use, or 10 ml. of a weak sample. It should be noted that the B.P. does not use normal temperature (0°) and pressure (760 mm. on the dry gas), but 15.5° and 747 mm.—that is, 760 corrected for the tension of aqueous vapour at 15.5° (13 mm.). The calculation of the proportion of ethyl nitrite depends on these constants: 1,000 ml. of nitric oxide measured at 0° and 760 mm. dry, or 1075.2 ml. measured at 15.5° and 747 mm. moist, weigh 1.3402 gm.; also 30.008 gm. of nitric oxide are equivalent to 75.047 mg. ethyl nitrite.

$$\left. \begin{array}{l} 1 \text{ ml. of nitric oxide} \\ \text{measured at } 15.5^{\circ} \\ \text{and 760 mm. moist, or} \\ 747 \text{ mm. dry.} \end{array} \right\} = \frac{1.3402 \times 75.047}{1075.2 \times 30.008} = 0.003117 \text{ gm. ethyl nitrite.}$$

In an analysis, the temperature and pressure should be read; after subtracting the appropriate tension of aqueous vapour from the latter, the volume should be corrected to 15.5° and 747 mm. (see p. 110) and the above factor used.

Lowe has described a sample of the spirit which contained potassium nitrate; in a case of doubt the total solids of the spirit should be determined (*S.P.A.*, 1899, **24**, 87).

PROSECUTIONS. *Birmingham.* Water in excess 50 % and only a trace of ethyl nitrite. Fine £2 (1889 Report).

Nottingham. Practically devoid of nitrous ether. The defendant, who was a grocer, had kept it in a corked bottle in the window, where sunlight could fall on it. Fine 10s. 6d. (*F. & S.*, 1896, May 30).

London, North. Deficient of 65 % of ethyl nitrite. The sample had been sold from a white glass shop bottle about one-third full, exposed to full daylight. Fine £10 (*C. & D.*, also *F. & S.*, 1899, March 18).

London, Marylebone. 30 % of nitrous ether abstracted, only 1.29 % being present. The sample was put in a half-pint bottle, and divided between two other similar bottles, each bottle containing a little more than an ounce. An analyst for the defence gave evidence that by treating a sample of full strength in the same way the prosecution sample had been divided, the strength fell from over 6 volumes of gas to 4.32 volumes, or 20 %. In another experiment, 3 oz. were put in a 3-oz. stoppered bottle, then divided between three 1-oz. stoppered bottles, when the loss was only 5 %, and on repetition 3 %. The case was dismissed (*C. & D.*, 1899, April 1; *B.F.J.*, 1899, 116).

London, North. Ethyl nitrite 34 % deficient. A public analyst gave evidence for the defence that a loss of 6 % or 7 % might occur during dividing the sample. By extracting an ounce from a bottle with a pipette on six consecutive days, the strength of the spirit fell from 5.5 volumes to 3.4 volumes. The defendant was ordered to pay 12s. 6d. costs only (*P.J.*, 1900, June; *B.F.J.*, 1900, 203).

Dublin. Ethyl nitrite only 0.4 %. The third sample was sent to the Government analysts, who reported that owing to bad corking the sample had evaporated to such an extent that analysis was impossible. Case dismissed (*P.J.*, 1901, Aug., Sept.; *B.F.J.*, 1901, 275, 301).

Saxmundham. Ethyl nitrite 82 % deficient. The case was dismissed, as the analyst had not reported on decomposition (*P.J.*, 1901, Nov.).

London, Worship Street. The vendor was charged for selling an article of food from which 70 % of nitrous ether had been abstracted. The magistrate held that "abstraction" must be wilful, and that the prosecution was taken under the wrong section, and dismissed the case (*P.J.*, 1904, Nov. 5).

Croydon. Alcohol deficient 40 %. The defendant stated that the article had been diluted with proof spirit instead of rectified spirit, and that the alcohol was only the solvent or preservative. Fine and costs £3 4s. (*P.J.*, 1911, April 29).

Connah's Quay. Ethyl nitrite 52.6 % deficient. The grocer, who sold it, said it was part of 1 lb. she had bought eight years previously. Ordered to pay costs (*P.J.*, 1924, Feb. 9).

Lincoln. Ethyl nitrite 71 % deficient. The deficiency was attributed to evaporation owing to a defective stopper. Case dismissed (*P.J.*, 1926, Oct. 23).

London, Woolwich. Ethyl nitrite, 0.17 %, and therefore deficient in the ingredient to the extent of 90.25 %. The magistrate dismissed the summons, holding that the certificate was too vague (*B.F.J.*, 1907, 126).

London, Lewisham. Ethyl nitrite deficient 40 %. The inspector stated that the filling of the three bottles did not take more than twenty seconds. The defence suggested that 20 % of the ethyl nitrite present might be lost by pouring into an open vessel and back. The Public Analyst thought it was possible, but said the loss depended on the shape of the bottle, the temperature of the hand and other considerations. The case was dismissed (*P.J.*, 1929, Nov. 30 ; *B.F.J.*, 1930, 8).

Pershore. Ethyl nitrite 44 % deficient. Though it was suggested that loss of strength occurred during sampling, the vendor was fined 30s. (*P.J.*, 1931, Jan. 3).

London, Tower Bridge. Ethyl nitrite 0.805 % instead of 1.52 %. The Public Analyst stated that in the six weeks since taking, the sample had only lost $6\frac{1}{2}$ % more. Pouring a sample from one bottle to another caused no appreciable deficiency, and after pouring three times from a height, the deficiency was 5 %. The defendant stated that he had only opened the stock bottle five times before the sample was taken. He complained that the inspector left the cork out and used bottles too large for the samples. A chemist gave evidence that pouring from one bottle to another might cause a loss of 17 %. The magistrate considered that, though some loss might have occurred during sampling, it would not account for the deficiency. He criticised the statement in the analyst's certificate that "No change had taken place" when a change amounting to 43.5 % was alleged to have taken place. Fine £10 (*C. & D.*, 1931, Oct. 31 ; *P.J.*, 1931, Nov. 7).

AROMATIC SPIRIT OF AMMONIA. SAL VOLATILE

The directions for making this spirit of the 1885 B.P. were followed by those of 1898 and 1914, except that alcohol 90 v/v took the place of rectified spirit (88.7 v/v). The total alkalinity due to ammonia and ammonium carbonate is required to be equivalent to 2.16 w/v of ammonia (NH_3). The limits suggested for the new B.P. are:—Sp. gr. 0.890–0.900. Total alkalinity equivalent to 2.1–2.4 w/v NH_3 . CO_2 1.265–1.485 w/v, equivalent to 2.26–2.65 w/v $\text{N}_3\text{H}_{11}\text{C}_2\text{O}_5$.

ALKALINITY OF SAL VOLATILE (Fifty-five samples)

NH_3 , w/v	.	1.2, 1.4	1.78–	1.9–	2.0–	2.1–	2.2–	2.3–2.41	Total.
Percentage of samples	.	4	9	11	18	24	23	11	100

Several of the samples were low in quality; one vendor was prosecuted and several cautioned. It will be seen that two-thirds of the samples were between 2 % and 3 %. In some cases the deficiency in alkalinity may have been due to the use of effloresced ammonium carbonate.

AMMONIUM CARBONATE IN SAL VOLATILE (Fifty-five samples)

$\text{N}_3\text{H}_{11}\text{C}_2\text{O}_5$, w/v	1.2-1.5	1.6-1.8	2.1-	2.3-	2.4-	2.5-	2.7-3.1	Total.	
Percentage of samples	.	6	13	25	18	25	7	6	100

The 1914 official limits of composition are 2.35–2.51 w/v, and about half the samples are near those limits. The samples low in carbonate are not due to the use of an effloresced article, as keeping makes little difference in the percentage of CO_2 in the carbonate.

By subtracting the amount of ammonia contained in the carbonate from the total ammonia, the free ammonia is obtained.

FREE AMMONIA IN SAL VOLATILE (Fifty-five samples)

Free ammonia, w/v	.	.	.7–	1.1–	1.2–	1.3–	1.4–	1.5, 1.6	Total.
Percentage of samples	.	.	7	7	13	26	31	16	100

Calculation from the official figures gives the limits of free ammonia as 1.34–1.40 w/v, and about half the samples were in the neighbourhood of these limits.

Papers on the composition and analysis of sal volatile have been given by Thresh (*B.P. Conf.*, 1880, 564; *Y.B.P.*, 1883, 273), Gravill (*P.J.*, 1886, 445), Liverseege (*P.J.*, 1895, Feb. 23), and E. White (*P.J.*, 1900, Feb. 17).

Of the samples examined in England and Wales during 1907–13, 9.5 % were adulterated, and 6.5 % of those during 1920–30.

DETERIORATION. Some experiments by the writer on the alteration on keeping sal volatile are here given. (I.) The spirit

was kept in a 4-oz. corked bottle half full for ten months. (II.) It was kept in a 6-oz. stoppered bottle half full for seven months. (III.) 6 oz. was kept in an 8-oz. stoppered bottle, and $\frac{1}{2}$ oz. was poured out most mornings during two months and returned to the bottle. It will be seen that the losses in these conditions are small :—

DETERIORATION OF SAL VOLATILE

Experiment	(I.)	(II.)	(III.)
Earliest determination	February.	July.	January.
Latest	December.	February.	March.
Increase in sp. gr.	—	0.001	0.001
Decrease in alkalinity, NH_3 , w/v	0.11	0.04	0.15
„ ammonium carbonate, w/v	—	0.04	0.07
„ free ammonia, w/v	—	0	0.12

ANALYSIS. In the determination of the *sp. gr.* the co-efficient of expansion is 0.00043 for 1°F. between $55^\circ\text{--}65^\circ \text{F.}$ The *total alkalinity* is determined on 10 ml. using $\text{N}/2 \text{ HCl}$ and methyl red. To avoid volatilisation of NH_3 the spirit should be run into water mixed with part of the acid, say 20 ml. An alternative method is to mix with a slight excess of HCl , evaporate to dryness, dry the NH_4Cl , and weigh. The *carbonate* may be determined by the B.P. method, by Hepburn's method (p. 75), or by mixing 5 ml. with an equal volume of HCl (1 in 2) in a nitrometer and measuring the liberated CO_2 . The correction for the solubility of the gas in the liquid is 7 ml. at 15.5°C. , and 0.12 ml. for each 1°C. difference from that temperature (Liverseege, *opus cit.*). The volume is corrected to 15.5°C. and 747 mm. (dry) pressure (p. 110), and the factor to obtain w/v of $\text{N}_3\text{H}_{11}\text{C}_2\text{O}_5$ is 0.653. The *free ammonia* is obtained by subtracting from the total ammonia, the product of carbonate multiplied by 0.325. The determination of *alcohol* has been given previously (p. 352).

PROSECUTIONS. *Birmingham.* Deficient of 37 % of its strength. (The total alkalinity was 1.38 w/v, and the ammonium carbonate 1.21 w/v). Fine £3 (1890 Report).

Keighley. Ammonium carbonate $2\frac{1}{4}$ oz., strong solution of ammonia $6\frac{1}{2}$ fl. oz. per 8 pints, instead of 4 oz. and 8 fl. oz., respectively. The loss of ammonium carbonate was certified not to be due to volatilisation or other similar causes. Fine and costs £1 (*B.F.J.*, 1901, 211).

Sproatley. Ammonia deficient about 30 %. It was suggested that there had been three days' delay in analysing the samples, and that the samples should have been put in stoppered bottles. Costs (*P.J.*, 1903, May 8).

Stepney. Chloroform 2.5 %, alcohol deficient 10.1 % (sp. gr. 0.930). Fine £5 (*Analyst*, 1926, 51, 514). Spirit of chloroform may have been used for making it instead of alcohol.

ESSENCE OF PEPPERMINT

The 1885 B.P. contained an "Essence of peppermint" 20 v/v of the oil, and also a "Spirit of peppermint" 2 v/v. The 1898 and 1914 B.P. gave only one preparation, "Spirit of peppermint" 10 v/v. In 1905 the *Chemist and Druggist* asked its readers what they sold as "Essence of peppermint," and received a number of replies, which named 6-20 v/v. The Editorial remark was, "Apparently the 10 v/v has it. The object in removing the strong essence from the B.P. and strengthening the weak spirit was, we understand, to provide one preparation equally suitable for prescribing purposes and retail" (Feb. 18, 25). The B.P. Codex gives "Essence" as a synonym for "Spirit," a course which is to be followed in the new B.P.

Randall (*Analyst*, 1924, **49**, 440) has given a method of determination, and Reilly, Noonan and Drumm have given a method for the evaluation of menthone in peppermint oil (*S.P.A.*, 1931, **56**, 702).

PROSECUTION. *Sunderland.* Oil of peppermint 0.3 %, alcohol 1.81 %, water 98.16 %, instead of 10 % of oil of peppermint in alcohol according to the B.P. For the defence it was contended that there was no standard for the "Essence," which was not in the B.P., and that if the B.P. "Spirit" had been sold the cost would have been 2s. 6d. instead of 7½d. Case dismissed (*Analyst*, 1927, **52**, 282; *P.J.* and *Grocer*, 1927, March 19).

COMPOUND TINCTURE OF BENZOIN

There has been a considerable discussion as to the amount of bark and other matter insoluble in 90 v/v alcohol that should be allowed in medicinal benzoïn. Southall's Laboratory Reports have indicated that about 70 % of Sumatra benzoïn (the only kind recognised by the B.P.) is soluble. Moor and Priest (*S.P.A.*, 1901, **26**, 32) found 9.2-16.7 % of insoluble matter in three qualities. Holmes (*P.J.*, 1907, Feb. 9) gave figures from which he concluded that first-class Sumatra benzoïn may contain as little as 4.5 % of impurity, second quality from 8-10 %, and inferior as much as 23 %. The amounts of ash present varied 0.6-2.4 %. A sample of Sumatra examined by the writer had 9.0 % of insoluble matter, 0.6 % of ash, and it lost 5.6 % on drying in the water oven. For a second quality the figures were 13.3 %, 0.8 % and 5.1 %, respectively. The 1914 B.P. prescribed a limit of 15 % of insoluble matter and one of 5 % of ash. Cocking (*Q.J.P.*, 1928, 338) considered the B.P. limit for insoluble matter was too severe, and that it should be raised to 20 %. Walmsley (*P.J.*, 1930, April 12) suggested a limit of 22 %, but as the highest ash in ten samples was 1.83 %, he considered the B.P. ash limit was much too high.

Six samples of "benzoin" bought in Birmingham in 1926 had 15.5-32.2 % of insoluble matter, 0.8-2.2 % of ash, and they lost 4.9-8.0 % in the water oven. These samples were passed as genuine, as no indication had been given that medicinal, and not industrial, benzoin was required. Sumatra benzoin contains cinnamic acid, and the total acids, expressed as benzoic, in four of the samples of Holmes (*opus cit.*) were 19.1-28.4 %.

In 1901, as a result of the Government analysts passing a sample of compound tincture of benzoin (friars' balsam) as genuine, Hill and Liversidge examined the constituents of the tincture and advocated a standard of 18.0 w/v of solid extract (*S.P.A.*, 1901, **26**, 283). Other workers have agreed with this standard—*e.g.*, Barclay and Mann (*P.J.*, 1902, March 15), H. W. Gadd (*P.J.*, 1904, Feb. 27). E. A. Cripps (*P.J.*, 1909, Nov. 20). Barclay and Cripps (*opus cit.*) gave a method for the determination of the balsamic acids present in the tincture, and Cocking (*Q.J.P.*, 1928, 337) has also given one, and also details in reference to the acid and ester values, and the determination of the total solids by drying *in vacuo* over sulphuric acid.

No fewer than eleven of the sixty-three Birmingham samples examined during 1890-8 were reported adulterated, and there were several prosecutions. One sample had only 6.2 w/v of solid extract, and another had 25 w/v of glycerin. During 1900-29 only six of the ninety-seven samples examined were adulterated; a fine of £20 inflicted in 1900 probably promoted this improvement. The following three tables are based on these ninety-seven samples. The solid extract was determined by evaporating 5 ml. in a 3-in. diameter flat bottom metal dish and drying in the water oven for three hours :—

Solid extract, w/v	11.5	14.2	14.7	16	17	18	19	21.0	22.6	Total.
Samples	3	7	7	29	35	13	3	97		

The following was the range of specific gravities, the co-efficient of expansion for 1° F. being 0.0005 :—

Sp. gr.	0.880	0.890	0.895	0.900	0.095-0.911	0.915	0.926	Total.
Samples	9	7	31	27	21	2	97	

The approximate alcoholic strength, determined as is given below, had the following ranges :—

Absolute alcohol, w/v	54.8	56.8	57	58	59	60	61	62-63.1	Total.
Samples	2	4	18	23	34	7	9	97	

The two samples which had the lowest proportion of alcohol were the same as those which had the highest solid extract and specific gravity; probably they had become concentrated by evaporation of alcohol.

Of the samples of friars' balsam examined in England and Wales during 1920-30, 3.6 % were reported adulterated.

Specific Gravity and Spirit. The specific gravity of a tincture depends on two factors which act in opposite directions. It is greater in proportion to the amount of solid extract, and less as the quantity and strength of the spirit increases. Advantage was taken when preparing the 1898 tincture to determine the increase in specific gravity produced by 1 w/v of solid extract, and to apply the factors obtained to ascertain the approximate proportion of absolute alcohol.

The spirit used in preparing the tincture had a specific gravity of 0.8342, and the resulting tincture contained 18.2 w/v of solid extract and had a specific gravity of 0.8960. These figures show that 1 w/v of extract increased the specific gravity by 0.0034, which is about $\frac{1}{300}$ of the extract. Further, 100 ml. of the tincture contained 89.6—18.2, or 71.4 gm. of spirit of specific gravity 0.8342, or 85.6 ml. The extract, therefore, had a volume of 100—85.6 ml., or 14.4 ml., which is about 80 % of its weight. A previous experiment in making the 1885 tincture gave very similar factors. On these results the following formulæ are based :—

Sp. gr. of spirit in tincture = Sp. gr. of tincture — extract/300.

Volume of spirit in tincture = 100 — (extract \times 0.8).

In practice, after correction of the specific gravity for the extract present, the strength of the spirit is obtained from the alcohol table (p. 562), and the proportion in the tincture calculated from its volume.

PROSECUTIONS. *Birmingham.* Excess water 20 %, glycerin 25 %, and deficient of 65 % of the proper ingredients (solid extract was 5 w/v). Fine £1 (*C. & D.*, 1898, 52, 343).

Birmingham. Only 64 % of the solid extract contained in the B.P. tincture (= 11.5 w/v). Fine £20 (*C. & D.*, 1901, Jan. 26 ; *B.F.J.*, 1901, 101).

Birmingham. Only 82 % of the solid extract contained in the B.P. tincture (= 14.7 w/v). The Government analysts reported that there was no evidence of deficiency, and the case was withdrawn and the defendant allowed one guinea costs (*P.J.*, 1901, July and Aug. ; *B.F.J.*, 1901, 243, 274).

Bridlington. Deficient of 13 % of solids, calculated on 18 w/v. Fine £1 (*P.J.*, 1920, May 8).

Hull. Absolute alcohol 46.2 %, instead of 59.5 %, and 21.1 % of total extractive matters, instead of 18 %. Fine £2 2s. (*P.J.*, 1929, July 27).

TINCTURE OF MYRRH

The B.P. 1914 requires the matter soluble in 90 v/v alcohol present in 20 gm. to be contained in 100 ml. Myrrh is required not to exceed 70 % insoluble in that alcohol. This requirement is not unreasonable, as twenty-two samples of myrrh and powdered

myrrh examined by Merson (*P.J.*, 1900, Jan.) yielded 33–48 % of soluble matter. The B.P. limit of 30 % soluble will include volatile oil and any moisture present ; if these amount to 4 %, the minimum amount of non-volatile matter in the tincture is 5.2 w/v.

The total solids in twelve Birmingham samples examined in 1928 varied 5.3–7.8 w/v ; eight of them were between 5.5 w/v and 6.4 w/v. The sp. gr. varied 0.851–0.864, and the alcohol 66.5–67.9 w/v ; the ash varied 0.013–0.045 w/v. Two other samples were condemned as they contained only 4.2 w/v and 4.3 w/v of total solids. The question arose as to whether their low solids were the result of careless keeping by the retail dealer, and experiments were made to test the effect of leaving bottles of tincture of myrrh uncorked.

About 2 oz. of a tincture containing 5.5 % of solid extract were kept in a 4-oz. bottle with a narrow neck. At the end of forty-nine days, evaporation had increased the solids to 8.7 %, and the sample was quite clear. After four weeks' longer keeping, there were a few insoluble particles, and after filtration the solid extract amounted to 15.7 %.

A similar experiment was made with an 8-oz. bottle, about half full of a weak tincture. At the commencement 4.1 % of solid extract was present. Three months afterwards it had increased to 5.3 %. At the beginning of the period 66 % of alcohol was present and 63 % after keeping. These experiments show that if tincture of myrrh be kept in an uncorked bottle there is loss of spirit, and that there may be a very considerable increase in the proportion of solid extract before the spirit becomes too weak to retain it in solution.

ANALYSIS. The total solids were determined by evaporating 5 ml. in a 3 in. diameter German silver dish and drying in the water oven for six hours. The amount of alcohol may be obtained by calculation from the sp. gr. and total solids, as explained under "Alcohol" (p. 353). The value for *K* was found to be 0.003, and the volume of the extract to be 84 % of its weight. Three tinctures were made and the sp. gr. and total solids of them determined, as well as the sp. gr. of the spirits used in making them (*Liverseege, C. & D.*, 1896, April 18).

Sp. gr. of the spirit
in the tincture
(= S_1) $\left. \vphantom{\begin{array}{l} \text{Sp. gr. of the spirit} \\ \text{in the tincture} \\ (= S_1) \end{array}} \right\} = \left\{ \begin{array}{l} \text{sp. gr. of} \\ \text{the tincture} \end{array} \right\} - 0.003 \times (\text{w/v total solids}).$

From the sp. gr. table given in the Appendix (p. 562) the w/v of alcohol (A_1) corresponding to S_1 may be found, and correction made for the volume of the total solids made as follows :—

$$\text{w/v alcohol in tincture} = \frac{A_1 \times 100 - 0.84 (\text{w/v total solids})}{100}.$$

The solids of the tincture should be tested by the B.P. nitric acid colour test.

PROSECUTION. *Nottingham.* Alcohol deficient 42 %. Medical evidence was given that the deficiency of alcohol was not to the prejudice of the purchaser, and apparently the prosecution agreed. The case was therefore dismissed (*C. & D.*, 1897, June 19 ; *P.J.*, 1897, 536).

TINCTURE OF OPIUM, LAUDANUM

The early prosecutions for deficiency in spirit were probably due to the belief by some pharmacists that the official process of maceration in cold proof spirit was unsatisfactory, and that heating the opium with water, and the use of a weaker spirit, gave a better preparation.

That there was some justification for this belief is shown by the fact that the 1898 B.P. prescribed hot water and reduced the strength of the spirit. It has not always been noticed that the alcohol strength of the finished tincture is not 45 v/v, but a smaller proportion, owing to the variable proportion of water in opium.

The morphine strength of the 1885 tincture was about 0.75 % ; variation of 0.70–0.80 % was allowed in 1898, and in 1914 it was standardised to 1.0 %.

Papers on the determination of total alkaloids in opium have been given by Rakshit (*S.P.A.*, 1926, **51**, 491), and on the estimation of meconic acid by Annett and Bose (*S.P.A.*, 1922, **47**, 387). Rakshit has also pointed out that the “ morphine ” precipitated in the 1914 B.P. method of assay is contaminated with codeine (*S.P.A.*, 1931, **56**, 711).

Of the seventy-nine samples examined in England and Wales during 1906–8, only two were reported adulterated.

PROSECUTIONS. *Sheffield.* Only 56 % of the spirit, and only 23/75 of the morphine, contained in the B.P. preparation. Evidence was given that the term “ tincture of opium ” was understood to apply to the tincture of the B.P. The stipendiary magistrate dismissed the case on the ground that B.P. tincture of opium was not specifically asked for. On appeal, *White v. Bywater* (1887), this decision was reversed ; the Appeal Court held that the defendant was liable to conviction, although the purchaser had not specifically asked for tincture of opium prepared according to the recipe of the B.P. Tincture of opium must mean the article known in commerce as tincture of opium. The thing supplied was not tincture of opium of commerce, but an inferior article (*C. & D.*, 1887, May 21 ; *P.J.*, 1887, May 21, 28).

Nottingham. No morphia present. The chemist was asked to supply laudanum, and contended there was a distinction between that and tincture of opium. Fine £5 and costs. Another chemist,

whose article contained about one-third of the morphia required by the B.P., was fined £3 (*F. & S.*, 1892, Aug. 13).

Corn Mill Bridge. Alcohol 23.5 %, being about one-half of the amount that should be present if it had been prepared according to the directions of the B.P. The defendant explained that the sample, being taken from the bottom of a gallon jar in which it had been kept, was low due to evaporation. Fine 5s. (*F. & S.*, 1894, Dec. 15).

Nottingham. Proof spirit 59 %, being deficient of 41 % according to the B.P. formula. Case dismissed, as the Public Analyst had used the 1885 B.P. as standard instead of the 1898 (*C. & D.*, 1900, May 12).

Aberdeen. Morphine deficient about 16 %. Fine £5 (*P.J.*, 1906, April 28).

PAREGORIC, PAREGORIC ELIXIR, COMPOUND TINCTURE OF OPIUM

Paregoric contains opium, and may therefore only be sold by a qualified pharmacist. Unqualified vendors have been prosecuted under the Food and Drugs Act for the sale of an article containing no opium, or under the Merchandise Marks Act for "paregoric" free from opium. The use of an article of which the essential constituent is absent will have little effect, and, on the other hand, there is the risk that a consumer, used to the substitute, may take the same quantity of the genuine article with serious results.

Samples of paregoric were first taken in Birmingham in 1890, when four of the nine samples were either deficient in opium, spirit, oil of anise, or contained glycerin. During 1892-1913, 12 % were adulterated, but the sixteen samples bought 1915-29 were all genuine. The range of the sp. gr. of the latter was 0.915-0.923, though for eleven of them it was only 0.917-0.920. Benzoic and meconic acids were 0.46-0.55 w/v, and twelve of them only varied 0.49-0.53 w/v. Of this total, about 0.04 w/v is meconic acid. The non-volatile solids of paregoric are about 0.3 w/v.

Two samples of "paregoric substitute" examined in 1913 contained no opium; the sp. gr. were 0.847 and 0.841, respectively, and had 0.1 w/v of non-volatile solids.

During 1905-13, 9.4 % of the samples examined in England and Wales were adulterated, and 2.8 % of those examined 1920-30.

ANALYSIS. Evaporate 10 ml. of the tincture to dryness with 2 ml. $\text{E.Na}_2\text{CO}_3$, add water and filter into a separator. Add 3E.HCl in excess (about 10 ml.), and shake out successively with 15, 10, and 10 ml. of ether. Weigh the residue after spontaneous evaporation, put in a desiccator for a day and weigh again. Treat with water, and titrate with N/10 NaOH, using phenol phthalein

The difference between the two methods was 0.04 w/v. Alcohol is conveniently determined by the Carl Jungk method (p. 353). The addition of 2 ml. of the tincture to about 50 ml. of water should give a decided milkiness; 0.2 w/v of oil of anise is almost clear. Subsequent addition of a drop of E/5. FeCl_3 solution should give a brownish-red colour, due to meconic acid.

Methods for the determination of morphine have been given by Bird (*B.P. Conf.*, 1905, 459), Caines (*B.P. Conf.*, 1927, 506), and Brindle (*P.J.*, 1927, Dec. 3).

An early paper on the analysis of paregoric was given by Allen (*S.P.A.*, 1879, 4, 104), and later he and Scott-Smith discussed the effect of ipecacuanha on the analysis (*S.P.A.*, 1902, 27, 350). A paper by Dovey (*S.P.A.*, 1927, 52, 26) is on the colorimetric determination of opium, and the distinction between meconate, acetate and thiocyanate of iron. Chartier (*Y.B.P.*, 1926, 289) describes a method for the determination of camphor, which, however, requires 40 gm. of paregoric.

PROSECUTIONS. *Sheffield.* Alcohol 34 % instead of 48 %, with only traces of benzoic acid and oil of anise. Fine £5 (*Analyst*, 1879, 4, 96).

Birmingham. Glycerin 30 %, and contained neither alcohol nor oil of anise. Fine £2 (1890 Report).

Birmingham. Water 50 %. Fine £3 and costs (*F. & S.*, 1893, June 10).

Beverley. No opium and only a small percentage of alcohol. The bottle from which the sample was taken was labelled "Paregoric Elixir without Opium." Fine 5s. (*P.J.*, 1906, June 2).

Barnsley. Entirely deficient of tincture of opium, and only half the alcohol required by B.P. The unqualified assistant labelled the bottle "Paregoric Substitute," the latter word being in pencil, and the word "Poison" having been crossed out. The purchaser saw the label put on, asked no question, and received the bottle wrapped in opaque paper. The defendant gave evidence that there was practically no difference in the value of B.P. paregoric and paregoric substitute. The magistrates, following the case *Jones v. Jones* (1894) decided that there had been no prejudice to the purchaser and dismissed the summons. On appeal, *Bundy v. Lewis* (1908), the High Court held that the action of the magistrates was justified (*P.J.*, 1908, May 2, Oct. 17).

Birmingham. No tincture of opium. The bottle was labelled "Paregoric Essence." Fine £1 (1913 Report).

Walsall. Applying a false trade description, "paregoric," and selling the liquid to which it had been applied. The liquid was destitute of opium. The defendant, who was not qualified, was fined £1. The prosecution was undertaken by the Pharmaceutical Society (*P.J.*, 1909, May 29). There was a similar case at Blackburn

(*P.J.*, 1924, Oct. 11; *Analyst*, 1924, **49**, 583; and at Coventry, where the vendor was fined £10 (*P.J.* and *C. & D.*, 1932, March 19).

PROSECUTION FOR PAREGORIC AND SYRUP OF SQUILLS.

Fulham. Morphine absent. Costs paid (*P.J.*, 1913, April 19; *B.F.J.*, 1913, 78).

TINCTURE OF RHUBARB

The "tincture of rhubarb" of the 1885 B.P. was made with proof spirit; the 1898 B.P. changed the name to "compound tincture of rhubarb," included 10 v/v of glycerin, and increased the strength of the alcohol to 60 v/v; the 1914 edition reduced the spirit for making it to 45 v/v; in the 1932 B.P. alcohol 60 v/v is again to be used. In each case cardamon and coriander were present, and 100 parts of the tincture corresponded to 10 of rhubarb. In addition to the glycerin, it contains about 5 w/v of solid matter. Partridge has given analyses of samples bought in 1919 (*S.P.A.*, 1919, **44**, 369). About the years 1894-8 there were numerous prosecutions for the tincture; during 1905-13, 3.6 % of the samples examined in England and Wales were adulterated, but the 301 samples of 1921-30 were all reported genuine.

PROSECUTIONS. *Birmingham*. Tincture of rhubarb 82 %, spirit and water 18 %. It was said to be the compound tincture of the London Pharmacopœia. Fine £3 (1894 Report).

Extractive 8.6 %, absolute alcohol 70.9 %, instead of about 5 % and 46 %, respectively. Fine 10s. (*C. & D.*, 1895, 487).

Chorley. Total solid matter, mainly extract of liquorice and sugar, 5.0 %, alcohol 23.7 %. It contained an excess of 40 % of water, and was almost devoid of rhubarb. The grocer who sold it was fined £5 (*F. & S.*, 1897, Aug. 7).

Skipton. Glucose 8.9 %, alcohol 31.4 %, instead of 49 %. Fine £1 (*F. & S.*, 1898, Jan. 8; *P.J.*, 1898, Jan. 8).

TINCTURES OF QUININE

There are two tinctures of quinine, and prosecutions have taken place for the substitution, perhaps accidental, of the one for the other. The simple tincture is a 2 w/v solution of quinine hydrochloride in tincture of orange, which is made from 90 v/v alcohol. The simple tincture costs about five times as much as the ammoniated tincture.

Ammoniated tincture of quinine is made from 2 gm. of quinine sulphate, 90 ml. of 60 v/v alcohol, and 10 ml. of solution of ammonia (9.6 w/v NH_3), and the volume of the mixture is 100.9 ml. (Haycock, *P.J.*, 1910, May 7). It therefore contains 1.98 w/v of quinine sulphate and 0.95 w/v of NH_3 , if there is no loss during preparation. In the new B.P. the volume is to be made up to 100 ml. An experiment by Rae (*P.J.*, 1929, Oct. 5) indicated a loss of 0.017 %

of NH_3 by filtering with an *open* funnel, but there was no loss if the funnel was covered. He also tested the loss on keeping. When 6 oz. were kept in a glass stoppered bottle, and occasionally opened, the loss of NH_3 in eight weeks was 0.011 w/v, and 0.045 w/v from a corked bottle in the same conditions. In another experiment 10 ml. were poured out daily from a 40-oz. bottle, properly stoppered. In eight weeks the loss of NH_3 was 0.045 w/v, and the amount of alcohol was unchanged (see also Cripps, *P.J.*, 1907, 519, and *Analyst*, 1929, 54, 419).

AMMONIA IN AMMONIATED TINCTURE OF QUININE, 1921-30
(Seventy-eight samples)

Percentage of NH_3 , w/v	·77-	·80-	·85-	·90-	·95-	1·00-	1·05-	1·23	Total.
Percentage of samples.	4	9	12	20	28	19	8		100

Besides these, six samples were low in ammonia (0.62-0.70 w/v). Two of them, from one vendor, were also notably deficient in quinine and alcohol. The table shows that the standard used (0.8 w/v) was sufficiently low to allow for any loss in reasonable keeping of the tincture.

The range of other figures obtained with the seventy-eight Birmingham samples was as follows :—

SP. GR.	·917-	·923	·924	·926	·928	·930	·932-	4	Total.
Percentage of samples	3		14	27	36	14	6		100
QUININE SULPHATE, w/v	1·80-	1·90-	1·95-	2·00-	2·05-	2·10-	2·20-	2·39	
Percentage of samples	6	13	13	23	19	18	8		100
TOTAL SOLIDS, w/v	1·64-	1·70-	1·75-	1·80-	1·85-	1·90-	1·98		
Percentage of samples	13	24	27	26	4	6			100

It will be seen that about two-thirds of the samples contained more than 2.0 w/v of quinine sulphate. The reason is that quinine sulphate ($\text{C}_{20}\text{H}_{24}\text{N}_2\text{O}_2$)₂, H_2SO_4 , $7\frac{1}{2}\text{H}_2\text{O}$, loses moisture on exposure to air, and so 2 gm. of such a partially dried substance will give too much quinine to the tincture. The theoretical quantity of moisture is 15.3 %, but eight commercial samples of quinine sulphate contained only 3.7-12.0 %. Loss of moisture ceases when the moisture is reduced to about $2\text{H}_2\text{O}$, or 4.1 % (Liverseege, *B.P. Conf.*, 1924, 752).

During the years 1912-16 no less than twenty-one of the forty-four Birmingham samples were condemned, but in 1921-30 the proportion was reduced to 12.5 %. Of the samples examined in England and Wales during 1905-13, 10.4 % were condemned, and 4.7 % of those examined 1920-5.

ANALYSIS. As B.P. quinine sulphate is neutral to methyl red, the NH_3 can be directly determined by titrating 5 ml. with N/10 HCl and that indicator. Quinine itself (Qu) is neutral to phenol phthalein, while the bisulphate (QuH_2SO_4 , $7\text{H}_2\text{O}$) is neutral to methyl orange. Upon these facts Seaton and Richmond (*S.P.A.*,

1890, **15**, 42) have given a method of determining quinine by titrating between the two indicators with N/10 Ba(OH)₂. See also Allen (*S.P.A.*, 1896, **21**, 85). The method is, however, hardly delicate enough for the quantities of tincture usually available, and weighing the alkaloid is better. Transfer the liquid used for the ammonia titration to a separator, add 5 ml. 18E.NH₄OH, and shake out with successive quantities of 25, 10, and 10 ml. of redistilled ether. Distil off the ether and dry in the water oven for four hours, or longer if necessary. Drying at 110° C. is unnecessary. Multiplication of the weight of the residue by 27.2 will give w/v of B.P. quinine sulphate. The *total solids* should be determined by evaporating 5 ml. in a porcelain dish, and drying two hours or more in the water oven. Multiplication of the total solids by 1.18 will usually give w/v B.P. quinine sulphate. The residue should be tested qualitatively for quinine.

According to J. Grant (*S.P.A.*, 1931, **56**, 653), quinine may be titrated in ultra-violet light, without addition of indicator. Walton and O'Brien have given a colorimetric method for the determination of quinine (*S.P.A.*, 1931, **56**, 714).

PROSECUTIONS FOR TINCTURE OF QUININE. *Greenwich.* Quinine sulphate 0.67 %, other alkaloids and sulphates 0.66 %, instead of 1.83 % of quinine sulphate. The vendor was fined £5 for selling a drug which had been mixed so as to affect its potency (*Analyst*, 1880, **5**, 168).

Alcester. Ammoniated tincture. Fine 10s. (*P.J.*, 1908, Jan. 4).

Oswestry. Not more than 55 % by volume of absolute alcohol, instead of at least 72 %. Evidence was given that the alcohol had no medicinal value, and the case was dismissed (*P.J.*, 1911, Jan. 7).

PROSECUTIONS FOR AMMONIATED TINCTURE OF QUININE.

Marylebone. Quinine sulphate 80 % less than the proper quantity. An accident was said to have happened. Fine 5s. (*B.F.J.*, 1900, 264).

Bournemouth. Deficient in alcohol 5 %, and in ammonia 21 %, of the proper quantities. The ammonia was 0.80 %, and the absolute alcohol 42.95 %. The defendant attributed the deficiencies to evaporation during dividing and loss through the sealed cork. The magistrates dismissed the case, remarking on the advisability of using hermetically sealed bottles when sending volatile drugs through the post (*C. & D.*, 1903, Aug. 1 ; *B.F.J.*, 1903, 186).

Bakewell. Deficient in quinine sulphate 90 %, and in ammonia 60 %. Fine 30s. (*P.J.*, 1912, May 18 ; *B.F.J.*, 1912, 120).

London, Thames. Deficient in ammonia 15.8 %. The deficiency was attributed to loss during filtration and opening the bottle during dispensing, but rebutting evidence was given of experiments which did not support these statements. Fine £2 and 15 guineas costs (*Analyst*, 1929, **54**, 418).

TINCTURE OF IODINE

“Tincture of iodine” of the 1898 B.P. became “Weak tincture of iodine” in the 1914, with no change of composition. It had almost the same composition as the 1885 preparation “Tincture of iodine.”

It is a solution in spirit containing 2.5 w/v each of iodine and potassium iodide. The use of potassium iodide is two-fold: it prevents the precipitation of iodine on dilution of the tincture with water (the B.P. of 1864 ordered 1.25 w/v only, and it was found insufficient); it also promotes stability. The acidity of a sample examined by Knoll (*Q.J.P.*, 1929, 142), made with 80 % alcohol only, after six months was equal to 0.84 % HI, while a similar sample with 1.75 % potassium iodide had an acidity of 0.06 %. In the 1932 B.P. the amount of potassium iodide is to be reduced to 1.5 w/v.

As alcohol is more volatile than iodine, the effect of keeping the tincture is to increase the proportion of iodine as well as the potassium iodide. On keeping a standard tincture fifty-eight months in a stoppered bottle, the iodine increased 0.13 w/v, the potassium iodide 0.14 w/v, while the alcohol decreased 0.4 w/v. Another sample kept in a corked bottle increased 0.07 w/v of iodine in twenty-six months, and a third sample gave identical analytical figures after thirteen months. In each case the bottles were kept in the dark (Liversege, *C. & D.*, 1899, April 29).

The following figures relate to eighty samples bought in Birmingham, 1900-29 :—

IODINE AND POTASSIUM IODIDE IN TINCTURE OF IODINE

Iodine, or potassium iodide, w/v	1.5-	2.4-	2.6-	2.8-3.16	Total.
Iodine, percentage of samples	10	64	15	11	100
Potassium iodide, percentage of samples	7	56	26	11	100

ABSOLUTE ALCOHOL IN TINCTURE OF IODINE

Absolute alcohol, w/v	64.5-	67-	68-	69-	70-70.7	Total
Percentage of samples	6	12	28	49	5	100

SP. GR. OF TINCTURE OF IODINE

Sp. gr.	.873-	.875-	.885-	.895-.906	Total.
Percentage of samples	6	75	16	3	100

There have been a number of fines in Birmingham for adulterated tincture of iodine, and there has been a marked diminution in the percentage of adulteration. During 1890-8, 31 % were adulterated; during 1900-11, 18 %; and during 1914-29, 12 %. For England and Wales, the figures were—1903-13, 10.7 %, and 1920-30, 7.9 %.

ANALYSIS. In 1894, the writer communicated a paper on the

analysis of tincture of iodine, and gave analyses of a number of samples (*B.P. Conf.*, 1894, 475).

As the reaction between *iodine* and starch is so delicate, a solution of $\text{Na}_2\text{S}_2\text{O}_3$, $5\text{H}_2\text{O}$, weaker than N/10 is satisfactory. A strength which simplifies calculation is 1.37 w/v, of which 1 ml. is equivalent to 0.007 iodine. *Potassium iodide* may be determined by evaporating 5 ml. to dryness several times with water in a porcelain dish, which removes the iodine, drying in the water oven and weighing. One sample yielded a deliquescent residue, due to the presence of 1.8 w/v of sodium iodide. The reason was that the mother liquor of potassium iodide had been used instead of the pure salt. In another sample 1.5 w/v of potassium acetate was detected.

The dried residue may be dissolved in water and titrated with AgNO_3 solution, or, alternatively, a solution of the tincture in water, from which the iodine has been removed by boiling, may be used.

For the determination of *alcohol*, distillation after addition of soda is not satisfactory, as iodoform contaminates the distillate; thiosulphate is better, though a little sulphur may come over. Cameron (*S.P.A.*, 1902, **27**, 87) advocated the addition of iron turnings and Alcock (*P.J.*, 1904, Jan. 2) shaking with mercury, which should produce a nearly colourless solution, but in the presence of methylated spirit it will be dark yellow. The liquid is made alkaline before distillation.

The indirect method, by calculation from the solids, previously given (p. 353), may be applied to this tincture. By dissolving iodine in spirit in a stoppered sp. gr. bottle, with nine marks on the neck, it was found that 1 w/v of iodine increased the sp. gr. by 0.0081, and potassium iodide by 0.0079. "K" was therefore taken as 0.008, as they should be in equal proportions. Using the formula previously given, when $0.008 = K$:—

$$\text{Sp. gr. of the spirit in the tincture } (= S_1) = \left\{ \begin{array}{l} \text{Sp. gr. of the} \\ \text{tincture } (= S_2) \end{array} \right\} - 0.008 (\text{w/v I} + \text{w/v KI}).$$

From the alcohol table given in the Appendix (p. 562), the w/v of alcohol (A_1) corresponding to S_1 may be found, but as iodine and potassium dissolved in spirit increase its volume, this figure will require reduction to give the alcohol in the tincture (A_2). The increase in volume of spirit produced by dissolving iodine or potassium iodide is 0.24 ml. per gm. The corrected figure will be :—

$$\text{w/v of alcohol in tincture } (A_2) = \frac{A_1 \times 100 - 0.24 (\text{w/v I} + \text{w/v KI})}{100}.$$

PROSECUTIONS. *Brentford.* Free iodine absent, being decolourised tincture of iodine. The defendant had labelled it "Prepared according to the British Pharmaceutical Conference,"

but not "White iodine." The magistrates dismissed the case as they considered the mistake had not been fraudulent (*F. & S.*, 1895, Jan. 26).

Birmingham. Iodine 6·2, potassium iodide 3·8, and glycerin 10 gm. per 100 ml. For the defence it was incorrectly stated that the article was "liniment of iodine." Fine £1 (*F. & S.*, 1895, Dec. 21).

Birmingham. Iodine 1·9, potassium iodide 2·6, gm. per 100 ml. Fine £5 (1900 Report).

Birmingham. Iodine and potassium iodide, each 20 % in excess. The excess was attributed by the defence to the use of troy weight instead of avoirdupois weight. Fine £1 (*P.J.*, 1901, Jan.).

Portsmouth. Methyl alcohol at least 7 %, and a trace of acetone. The tincture had been made with methylated spirit. It had been put in a poison bottle and labelled "Iodine paint." The Bench cautioned the defendant and dismissed the case (*P.J.*, 1908, Dec. 19).

Hull. Alcohol deficient 12·5 %. Fine £2 (*P.J.*, 1912, May 18).

Marylebone. Excise prosecution under the Spirits Act, 1880, for making the tincture with methylated spirit. Fine £30 (*P.J.*, 1919, Oct. 4).

Ealing. Iodine 0·66 % in isopropyl alcohol, and no potassium iodide. Cartons labelled "Tincture of Iodine, B.P." were exposed for sale, but those actually sold were labelled "Iodine" in large letters, and "solution" below in small letters. For the defence it was stated that the change from genuine to substitute was made because the Customs and Excise Department objected to the sale of the former without an excise or spirit licence. The defendant's analyst found 0·67 % of free iodine and 0·53 % of combined iodine. Fine £2 (*P.J.*, 1929, June 22; *Analyst*, 1929, 54, 470).

Littledean. Iodine 18 %, and potassium iodide 14 %, deficient. The defendant proved a warranty and the case was dismissed. Subsequently the wholesaler was prosecuted for breach of warranty. The explanation given was that in its manufacture the lower layer, containing an excess of iodine, had not been mixed with the upper part. Fine £5 (*P.J.*, 1929, July 20).

STRONG TINCTURE OF IODINE

This preparation of the 1914 B.P., containing about 10 w/v of iodine and about 6 w/v of potassium iodide, dissolved in spirit, took the place of the strong solution of iodine of the 1898 B.P. In the 1932 B.P., a return is to be made to the name "solution."

In 1915, a prescription "Tinct. Iodi Fort" was dispensed at each of nineteen Birmingham pharmacies. One sample was apparently prepared of double strength, containing 19·5 w/v of

iodine, and 11.0 w/v of potassium iodide ; four samples contained 6.5–8.3 w/v of iodine, seven were about the right strength, 8.9–10.9 w/v, and seven samples contained 11.2–15.9 w/v of iodine. The proportion of potassium iodide was less variable ; two samples had 4.7–4.8 w/v, six samples had the correct amount 6.0–6.3 w/v, six had 7.0–7.7 w/v, and four had 8.0–8.5 w/v of potassium iodide.

For some of these unsatisfactory results, the explanation is carelessness in preparation ; some of the high figures are due to evaporation ; in others, the “ *Liquor Iodi Fortis* ” of the 1898 B.P. was dispensed ; though it was stated in the B.P. to be “ approximately ” the same strength, it contained about 11.7 w/v of iodine, being about 17 % stronger than the 1914 preparation.

PROSECUTION. *London, Old Street.* The weak tincture was supplied in error. Paid costs (*P.J.*, 1921, Jan. 8).

IODINE PAINT

There are various preparations of “ *Pigmentum Iodi* ” varying from 5–28 % of iodine, some containing glycerin. “ Iodine paint ” as sold by nine Birmingham pharmacists contained 2.2–3.0 w/v iodine, and one had 11.2 w/v. In 1924 the writer suggested that the label should, in addition to “ Iodine paint,” state the actual preparation supplied, such as “ Weak tincture of iodine ” (*B.P. Conf.*, 1924, 757).

CHAPTER XXXV

DRUGS IN DOSES—PILLS, CAPSULES, TABLETS, LOZENGES

Manufacture. Requirements:—Quantity, composition, coating. General analysis. Adulteration. Iron, carbonate of iron, and Blaud's, pills. Mercury, or blue, pills. Lead with opium pills. Ammoniated quinine capsules. Tablets:—Aspirin, acetylsalicylic acid. Phenacetin. Salol. Potassium chlorate. Sodium salicylate. Calcium lactate. Sodium citrate. Bismuth. Sugar and milk, with coffee or tea. Lozenges:—Chlorodyne. Bismuth. Compound bismuth. Tannic acid.

MANUFACTURE. At the dispensing counter pills are usually rolled in magnesium carbonate to prevent them sticking, but talc (French chalk) and ground liquorice are used. They are sometimes varnished, silver-coated, pearl-coated with talc, or other special coatings may be used. On the manufacturing scale, pills are made by piping presses and cutting machines, and are pearl- or sugar-coated in rotating spherical copper pans. They are also gelatin-coated.

Tablets are made in machines which have a cavity of adjustable size which is automatically filled with the material, after which the die descends and forms the tablets by compression. In some cases the drug is mixed with talc, starch, sugar, etc., so that it may flow uniformly through the hopper of the machine, and also to promote cohesion of the material into a firm tablet. It is necessary that the mixture should be in a uniform state of division, or equal volumes will not correspond to equal weights, and therefore some tablets will weigh more than others. For tablets to be satisfactory, it is necessary that, in addition to the powder being uniform and the machine working satisfactorily, the cavity shall be adjusted so as to contain a volume which shall represent the correct weight of the drug, otherwise the tablets may be uniform, but uniformly contain too much or too little of the drug.

A report by Davis (*B.P. Conf.*, 1891, 490) on the lozenges of the 1885 B.P., from different makers, showed considerable variation in size, weight and strength. Subsequent work on those of the 1898 B.P. (*B.P. Conf.*, 1899, 464) indicated a satisfactory state of commercial lozenges.

REQUIREMENTS. In the examination of these articles there are three things to be considered. (1) Does the quantity of the drug correspond with what has been demanded? (2) Is the drug supplied of the correct composition? (3) Is the coating, or other

addition, if any, suitable, and free from interference with the disintegration of the article ?

(1) QUANTITY. As these articles are made in various weights, a purchaser should ask for a definite weight—"5-grain iron pills," for instance, not merely "iron pills;" otherwise the analyst has no definite standard of comparison. In such a case each pill should contain 5 grains of the iron pill mass of the B.P., apart from the weight of any coating. Some allowance must be made for variation, as it is impossible to make all the pills of a batch exactly the same weight. In a coated pill there is no standard for total weight, and comparisons must be made with the average weight of a number of the pills. An allowance of 10 % under or over the mean weight appears to be liberal for 5-grain pills. The result of weighing more than 700 iron pills of that weight, taken from nineteen samples, comprising pearl, sugar, and gelatin-coated pills, from various makers, was as follows:—77 % were within 5 % of the mean weight, 19 % were between 5–10 %, and 4 % were more than 10 % from the mean weight. The examination of some pills indicated that the variation was due more to the coating than to the pill mass. One sample, of which 37 % of the pills differed 5–10 % from the mean, and 10 % more were over 10 % out, was inexcusable.

Frequently the box or bottle, containing the pills, has a label stating the weight of the contents. Serious variation from the stated composition renders the vendor liable to prosecution for false label under sect. 30 (1) of the 1928 Act.

Tablets were found to be more uniform than the above pills. More than 2,000 individual tablets, from fifty-seven samples, of six different drugs, were weighed 1925–7. 91 % of them were not more than 5 % above or below the mean weight of the tablets of the sample, 8 % were 5–10 % from the mean, and only 1 % exceeded 10 %. Unsatisfactory samples, eight in number, are not included in these averages. One sample of salol tablets had only 22 % under 5 % difference from the mean, 40 % were 5–10 % out, 25 % were 10–15 % out, and 13 % were over 15 % different from the mean !

When the total weight is under 5 grains, one expects the percentage error to increase, but with 2-grain sodium citrate tablets the increase was only slight. Over 600 individual tablets from nine different samples (all) were weighed, and 86 % of them were less than 5 % from the mean weight, 13 % were 5–10 %, and only 1 % were more than 10 % out.

(2) COMPOSITION. Not only must the drug present in pills, etc., be of the correct weight, but its composition also must be satisfactory. For example, an iron pill might contain exactly 5 grains of iron pill mass, but the mass might not contain the requisite 20 % of ferrous carbonate. Again, the aspirin in a tablet might contain an undue excess of salicylic acid, or talc might be present *in*

a pill mass, as well as in the coating. Various methods of analysis are given later.

(3) COATING. According to Rose (*Y.B.P.*, 1924, 378) less than one-half of the samples of "chocolate-coated" pills and tablets contained chocolate.

While the use of talc as a coating is probably harmless to adults (cp. Parkes and Roberts, *S.P.A.*, 1911, **36**, 389), its use appears to be undesirable in tablets used for infants' milk, such as sodium citrate, a sample of which had 3.8 % of talc. The amount of coating used should be moderate; satisfactory sugar-coated iron pills weighed 6.4 grains, while others weighed 9 grains.

It is possible that coating may be so thoroughly done that the pill or tablet passes through the body unchanged. Pills may be readily tested for rate of disintegration, by putting two into a 4-oz. round bottle about half full of cold water, lying the bottle on its side, and giving an occasional shake. In these conditions the sugar coating of an iron pill may be removed in five minutes, and black and white pearl coating in an hour, or less, while gelatin-coated pills may be considerably swollen in two hours. If the removed coating is then washed away, the action of the water on the inner coat and the contained pill mass is more readily observed. Within twenty-four hours an iron pill should be completely broken down, though not dissolved. In some cases the inner coat is almost impervious, due to the pills being varnished and then dusted with talc, before the main coat is added. Gronberg has conducted a research on the disintegration of pills (*P.J.*, 1920, Feb. 28).

When tablets are placed on the surface of cold water, many of them dissolve or break down into a powder within a minute or two. Some tablets are taken for the relief of pain, and therefore a rapid breaking down is desirable. Some tablets when placed in water remain unchanged for days, but chemicals may dissolve out and leave a skeleton of starch or talc closely resembling the original tablet. They may also be tested in the same way as pills. As a general statement, the tablets containing the largest proportion of talc were the slowest in disintegration. Kuever (*P.J.*, 1928, July 7) has discussed the solubility and disintegration of American tablets.

The B.P. Codex defines two varieties of talc: one is described as "French chalk," and the other, which has been treated with acid, as "purified talc"; it resembles the "powdered talc" of the B.P. The purified article should be used for pills and tablets. French chalk varies considerably in composition, the acid soluble part containing lime, which, expressed as chalk, in eight samples varied 1.0–10.6 %. The amount of arsenic found was trivial—0.4 parts per million.

GENERAL ANALYSIS. After weighing all the pills or tablets together to obtain the average, individuals should be weighed to

ascertain the proportion within the 5 % or 10 % variation. Two or more *average weight* pills should be taken for analysis, but tablets should be powdered and amounts weighed for analysis. Some experiments showed that the composition of heavier tablets was the same as of lighter ones, so that a heavy and a light tablet did not each contain the correct amount of drug, with more or less of the binding material.

ADULTERATION. In England and Wales, 5·8 % of the pills examined 1920–30 were reported adulterated, 4·2 % of the medicated lozenges examined 1920–5, and 6·6 % of the medicated lozenges and medicated tablets examined 1926–30 were condemned.

IRON PILLS, CARBONATE OF IRON PILLS

The 1914 B.P. requires that “iron pills” shall contain about 22·5 % of ferrous carbonate, but, according to the official description of the ingredients, it need not contain more than 20 % (Liverseege, *P.J.*, also *C. & D.*, 1927, Jan. 29). The “carbonate of iron pills” of the B.P. Codex may contain 30–40 % of ferrous carbonate. An important difference between these two pills is that while B.P. iron pills contain the ingredients of ferrous carbonate (ferrous sulphate and sodium carbonate), the carbonate of iron pills of the B.P. Codex contain that constituent ready made, and free, or nearly so, from the sodium sulphate which results from the decomposition. They are therefore different in composition, and one should not be substituted for the other.

About 1832 a French physician, Blaud, prescribed pills made from ferrous sulphate and *potassium* carbonate, and a similar pill was included in the 1890 Additions to the B.P., with the note that they were commonly known as “Blaud’s pills.” The 1898 B.P. altered the composition, retained the name “iron pills,” and omitted the note.

There has been carelessness in discrimination between the two articles ; in 1897, one of the five samples of carbonate of iron pills was iron pills, and on the other hand, seven of the twenty-seven samples of iron pills examined in 1926 were carbonate of iron pills.

A 5-grain pill should contain at least 1 grain of ferrous carbonate, and twenty-seven of the thirty-two samples contained 1·0–1·4 grains ; three samples were slightly low, containing 0·9 grain, another, probably a 4-grain pill sold in error, had 0·6 grain, and another only 0·3 grain.

There was a considerable variation in the total weight of each class of samples. The sugar-coated ones varied 6·4–9·0 grains per pill, the pearl-coated samples 6·3–8·3 grains, and the gelatine-coated pills 5·2–7·4 grains. A number of the pills, therefore, had more coating than was necessary.

The following are analyses of five typical pills (Liverseege,

opus cit.). "A" were sugar-coated pills, and therefore lost a considerable proportion on ignition. "B" were pearl-coated pills, as is seen by the amount insoluble in HCl. "C" were pearl-coated pills, labelled "Pil. Ferri, B.P.," but the low soluble ash and sodium sulphate prove the label to be a false one, as they were carbonate of iron pills. "D" were gelatine-coated pills with low soluble ash and sodium sulphate; they may have been made from a badly washed carbonate of iron, or from an iron pill mass from which part of the soluble salts had been removed by squeezing. "E" were pearl-coated pills, the pill mass of which was dark green in colour. The soluble ash (0.81) was much lower than the sodium sulphate, indicating a deficiency of carbonate, and that part of the iron was sulphate:—

IRON PILLS, WEIGHT IN GRAINS

	A	B	C	D	E
Iron oxide (Fe_2O_3)	0.70	0.76	1.02	0.86	0.90
Ash insoluble in HCl	0.06	1.34	1.75	0.18	1.96
Soluble ash (by difference)	1.17	1.26	0.27	0.38	0.81
Total ash	1.93	3.36	3.04	1.42	3.67
Loss on ignition	4.57	2.74	3.52	3.78	2.87
Total weight	6.50	6.10	6.56	5.20	6.54
Sodium sulphate (calculated from SO_4)	1.17	1.16	0.16	0.42	1.59
Ferrous carbonate (calculated from SO_4)	0.96	0.95	0.13	0.35	1.30
„ (calculated from Fe_2O_3)	1.02	1.11	1.49	1.25	1.31
„ (by titration)	1.00	0.89	1.42	1.23	1.20

Comparison of the last two lines of the table indicates that nearly all the iron was in the ferrous state, so that there had been little oxidation on keeping. One sample that had been kept in the laboratory fifteen months indicated 0.99 grain by titration, and 1.05 by the total iron.

The diameter of about 100 round pills was measured and found to vary from 6.8–9.0 mm.

The following are references to papers on iron pills:—Humphrey, on the origin of Blaud's pills (*P.J.*, 1903, May 9); Moerk, assay (*P.J.*, 1903, Aug. 22); Gough, assay (*P.J.*, 1903, Dec. 12); Greenish, stability (*P.J.*, 1904, Nov. 12); Parkes and Roberts, analyses (*S.P.A.*, 1911, **36**, 387); Parkes and Major, analyses (*P.J.*, 1914, May 23); Editor of *Chemist and Druggist*, origin and standards (1927, Jan. 29); and Abraham, composition of Blaud's pills (*P.J.*, 1927, Dec. 17).

During 1903–13, of the iron pills examined in England and Wales, 14.8 % were reported adulterated.

ANALYSIS. Cut or break two average pills, ignite at a moderate temperature till completely oxidised, which takes about half an hour, and weigh the total ash. Heat with strong HCl, dilute, filter, ignite and weigh talc. Precipitate the filtrate with AmOH, and weigh Fe_2O_3 , which is calculated to FeCO_3 .

Determine sulphate by heating four pills with dilute HCl, and dilute to 200 ml. After standing, decant and filter, and precipitate 100 ml. with BaCl_2 . Calculate to FeCO_3 , on the assumption (which may not be correct) that the SO_4 represents the ferrous sulphate, and that none of the SO_4 has been removed. Carbonate of iron pills obviously give low results for FeCO_3 calculated in this way.

For the determination of ferrous carbonate, weigh three average pills, put them in a mortar and cut or break them. Add 20 ml. of $3\text{E.H}_3\text{PO}_4$, allow to stand twenty minutes, periodically grinding to completely break down the pills. Add about 40 ml. of water and titrate with $\text{N}/10 \text{ K}_2\text{Cr}_2\text{O}_7$, using ferrocyanide as indicator (1 ml. $\text{K}_2\text{Cr}_2\text{O}_7 = 0.169$ grain FeCO_3). If on further standing an addition of more than a few tenths be required, the breaking down was probably incomplete and the determination should be completed (cp. Liverseege, *C. & D.*, 1897, Sept. 18). Diphenylamine as an internal indicator (Dyer and Forbes, *B.P. Conf.*, 1926, 416; *Analyst*, 1926, 51, 536) also gave satisfactory results when the sugar coating was previously removed. The solution must be standardised to this indicator.

Test for talc in the pill mass by washing off the coats with water, treating the kernel of the pill with dilute HCl, and igniting and weighing any residue.

PROSECUTIONS FOR IRON PILLS. *North London.* Deficient in 63 % of ferrous carbonate. The Government analysts gave a similar report. The pills were made abroad and bought without a warranty. Fine £5 (*C. & D.*, 1903, Feb. 21, March 14).

London, Lambeth. "Deficient in ferrous carbonate to the extent of 56 % of the total amount that should be present." The assistant told the inspector that they were "Blaud's Iron Pills." The certificate was objected to as giving no standard, and the Public Analyst stated that 8.8 % of ferrous carbonate was present instead of 20 %. He was unaware that Blaud had published his formula. The magistrate dismissed the case, considering that no one knew what Blaud's pills really were (*P.J.*, 1903, May 9).

London, Lambeth. Matter devoid of ferrous carbonate 100 %. The defendant had served pennyroyal pills. He stated that "Royal pills" were asked for, but failed to convince the magistrate, who fined him £1 (*P.J.*, 1903, May 16).

London, South-Western. Deficient in ferrous carbonate 66 %. Fine £10 (*P.J.*, 1903, July 4).

PROSECUTION FOR BLAUD'S IRON PILLS. *North London.* Ferrous carbonate 4.3 %, instead of 20 %. They were labelled "These pills are not prepared according to the formula of the British Pharmacopœia." The bottle was wrapped in paper, and the purchaser's attention was not called to the notice. The Public Analyst gave evidence that pills prepared according to the B.P., or

the French Codex, or Blaud's formula published in 1831, agreed in containing about 20 % of ferrous carbonate. When made the pills could not have contained more than 8.4 % of ferrous carbonate. The magistrate held that the pills should have conformed either to the iron pills of the B.P. or the original formula of Dr. Blaud. He also held that it was not a proprietary article, and that the label was not a satisfactory defence, as no evidence of mixture had been given, and the purchaser received the article wrapped up. Owing to previous convictions, the fine was £50 (*P.J.*, 1903, Aug. 15; *B.F.J.*, 1903, 206).

MERCURY PILL, BLUE PILL

According to the B.P., this pill should contain 33.3 % of mercury. When bought for analysis, pills of 4 grains, or a definite number of grains, should be specified, so as to give a definite standard. Obviously, a *coated* pill will not contain 33.3 % of mercury, though it has been correctly prepared.

PROSECUTIONS FOR BLUE PILLS. *Boottle.* Consisted of rhubarb pills. The defence was that the buyer was asked if he required a blue cathartic pill. Fine £1 (*C. & D.*, 1902, Dec. 20).

Boottle. Calomel about 1 grain per pill, and no metallic mercury. It was stated that Plummer's pills had been sold by mistake. Fine 10s. (*C. & D.*, 1902, Dec. 27).

Reigate. Mercury in each pill 0.50 grain, vegetable matter 1.87 grains, coating 1.32 grains—total, 3.69 grains. The percentage of mercury in the pill mass (excluding the coating) is 20.8, or 12.5 % deficient. They were bought as 3-grain pills. The Government analysts also analysed the pills and found 26.2 % of mercury. The pills were stated to have been sent out as received four days before the sale. Fine 1s. (*P.J.*, 1903, June 20).

Guildford. Mercury 17.4 %, vegetable matter 82.6 %. No definite size of pill was asked for. The Public Analyst stated that the average weight was about 6 grains, and if they had been sold as 3-grain pills, the only objection to them would be that they were rather large. The case was dismissed, as the vendor was not asked for any specific strength. The prosecutor asked for blue pills and got them (*P.J.*, 1903, July 11).

LEAD WITH OPIUM PILLS

PROSECUTIONS. *London, Mansion House.* Lead acetate absent, but mercurous chloride, sulphurated antimony, and other foreign ingredients were present. The prescription ordered "Pil. Plumbi cum Opio"; the dispenser said he misread "Plumbi" for "Plummer." The writing was quite legible. Three previous offences were proved, and the vendor was fined £10 and costs (*P.J.*, 1908, Nov. 14).

London, Guildhall. Lead acetate deficient, 16.9 %. The summons was dismissed as the certificate did not sufficiently disclose the particulars (*P.J.*, 1908, Nov. 21).

AMMONIATED QUININE CAPSULES

PROSECUTIONS. Capsules purchased in Liverpool labelled that each capsule contained about as much quinine as 1 drachm of the tincture were reported as containing no ammonia and an average $\frac{2}{3}$ grain of quinine sulphate. There was a considerable variation between the capsules in the amount of quinine. The Government analysts found a nominal amount of ammonia. The case was dismissed as they were not sold as "ammoniated tincture of quinine capsules" (*C. & D.*, 1905, Dec. 23).

A Birmingham sample claimed for each capsule "about one teaspoonful of ammoniated tincture of quinine." The amounts present were only about 60 % of those guaranteed, even when a teaspoonful was taken to be only 3.5 ml. The manufacturer attributed the deficiency to imperfect mixing, and the capsules were withdrawn from sale (1930 Report).

ASPIRIN, ACETYLSALICYLIC ACID, TABLETS

A number of Birmingham 5-grain samples were examined 1925-7, and in 84 % of them the average amount of aspirin varied 4.8-5.2 grains. The extremes were 5.6 and 4.6 grains, and the labels of these, which said "5 grains," were therefore false ones. In twenty-four of the twenty-six samples the free salicylic acid was 0.0-0.4 %, one was 0.5 %, and one 0.9 %. The formula in the B.P. Codex includes 2 % of talc, but only four out of twenty-seven samples approached this figure, having 1.9-2.3 %; and, on the other hand, sixteen samples were free from it. Excess of talc was present in five samples, the amounts being 2.6-5.7 %.

ANALYSIS. Two methods of *determination* have been found satisfactory; in either case the effect of any starch, dextrin, gum, or talc present is negligible. (1) Dissolve 1 gm. in 50 ml. neutralised methylated spirit, add phenol phthalein, and titrate with N/2 aqueous NaOH. (2) Add 25 ml. N/2 alcoholic NaOH to 1 gm. in a closed flask, and heat five minutes on the water bath. Cool, and titrate with N/2 HCl, until the colour ceases to return with shaking, phenol phthalein being used as indicator. The quantities thus determined should closely approximate to the amount soluble in ether.

Free Salicylic Acid. Dissolve 1.0 gm. of aspirin in 20 ml. of methylated spirit, dilute with water to 100 ml., and allow to stand for about half an hour to allow most of the starch to settle. Pour 30 ml. into one Nessler glass, and 10 ml. into another, adding 4 ml.

of methylated spirit, and in each case dilute with water to about 60 ml. Then add 1 ml. 1 % iron alum solution to each, mix and add standard salicylic acid to the second cylinder to match the colour of the first. The standard solution is made by diluting ten times a solution of 0.1 gm. salicylic acid dissolved in 5 ml. of methylated spirit and 95 ml. of water. As the difference between the two cylinders corresponds to 200 mgm. of aspirin, and as 1 ml. of the dilute solution contains 0.1 mgm. of salicylic acid, each ml. used indicates 0.05 % of free salicylic acid in the aspirin.

As aspirin contains no ash, the amount of ash found is probably due to talc; its solubility in acid should be tested. A test for starch should be made. Papers by A. N. Smith (*Analyst*, 1920, 45, 368, 412) deal with free acetic acid and tartaric acid in aspirin, and point out that the latter has a masking effect on salicylic acid.

PROSECUTION. *Hampstead.* A herbalist sold as "5-grain aspirin tablets" sixteen tablets which only contained about 4.4 grains each. For the defence it was pleaded that either the tablet machine was working irregularly, or that the loss was due to the tablets being kept in a cardboard box by the vendor. Fine £2 (*P.J.*, 1928, June 30).*

PHENACETIN TABLETS

The average weight per tablet of seven samples of 5-grain tablets bought in 1928 varied from 5.5–6.2 grains, and the average amount soluble in methylated spirit was 4.9–5.1 grains; the melting-points of the extracts varied 135–138° C. Starch was present in each case, and the ash (talc) varied from 0.1–3.0 %.

SALOL TABLETS

The average weight per tablet of six samples of 5-grain tablets bought in 1928 varied from 5.7–6.8 grains, and the average amount dissolved by ether (dried in desiccator only) was, for five of them, 4.8–5.4 grains. The sixth sample had only 4 grains. Three samples yielded no ash, the others had 4.7 %, 6.0 % and 12.2 %, respectively; the first of these disintegrated poorly in cold water, and the other two not at all. With the exception of these two, starch was present.

POTASSIUM CHLORATE TABLETS

Five samples of 5-grain tablets were almost pure chlorate of potash, and the average weight per tablet varied 5.1–5.4 grains. Two samples had 1.4 % and 1.7 % of talc.

SODIUM SALICYLATE TABLETS

The average weight per tablet of six samples sold as 5 grains, examined in 1925, varied from 6.0–6.1 grains, and the salicylic acid

* See Addenda, p. 578.

in them, calculated as 100 % (the B.P. requires at least 99.5 %), varied only 4.9–5.0 grains. Four samples were free from ash insoluble in acid; the other two had 5.2 % and 6.9 %, respectively.

ANALYSIS. If samples containing talc are ignited, combination occurs between it and the sodium carbonate formed from the salicylate, and interferes with subsequent analysis. When present it should be removed as follows:—Treat 2.2 gm. with water, filter through A.1, 9 cm. filter paper, and wash till filtrate measures 110 ml. Ignite and weigh any insoluble residue and test its solubility in dilute HCl.

Two methods of determination are available: (1) Evaporate 50 ml. of the filtrate to dryness, and ignite till fuming ceases. Heat ash with 70 ml. N/10 HCl, filter, wash, and titrate filtrate with N/10 NaOH, using methyl red. (2) Add 3 ml. 3E.HCl to 50 ml. of filtrate in a separator, and shake out successively with 20, 10 and 10 ml. of ether. The ether extract is weighed in a glass dish after standing two days.

CALCIUM LACTATE TABLETS

Calcium lactate, according to the 1914 B.P., should contain the equivalent of not less than 93 % pure calcium lactate, while the 1932 B.P. is to require at least 97 %. On drying, pure calcium lactate loses 29.2 % of water. The 5-grain tablets may therefore contain 3.29–3.54 grains of dry calcium lactate, and thirteen of the sixteen samples examined 1925–7 were within this range; the others averaged 3.6, 3.6, and 3.7 grains, respectively.

The average weight of the tablets in one sample was 6.7 grains, in ten samples the average varied from 5.1–5.9 grains, and in five it varied 4.5–4.8 grains. The low average weights of the last ones are not due to deficiency, but to the calcium lactate used having been partly dried. The moisture present in the sixteen tablets varied 18.9–27.5 %, ten of them being over 24 %. Several samples were free from talc, but one sample contained 7.0 %.

ANALYSIS. Talc, if present, should be separated in the manner given for sodium salicylate tablets; a little warming is helpful, but starch must not be gelatinised. Three methods of determination are available: (1) The same as sodium salicylate. (2) Take 50 ml. of the filtrate after removal of talc, evaporate and ignite. Add about 3 ml. 3E.H₂SO₄, evaporate and ignite. Repeat with 1 ml. more, ignite and weigh sulphated ash, which is calculated to anhydrous lactate. (3) Ignite 1 gm. of the tablets in powder, treat with water and HCl, filter, wash, add AmCl and AmOH to filtrate, filter, and determine calcium in filtrate. The amount of moisture in the tablets should be determined.

SODIUM CITRATE TABLETS

Sodium citrate was not contained in the 1914 B.P., and the amount of dry sodium citrate that should be present in 2-grain tablets is uncertain. The commercial salt may contain 2 molecules (12.3 %) of water or $5\frac{1}{2}$ molecules (27.7 %). The corresponding figures for the dry salt are 1.8 grains and 1.4 grains, respectively; and the eleven samples examined gave averages within these figures. The total weights varied 1.9–2.6 grains, and the moisture 0.1–13.9 %, showing the salt used was very variable. The 1932 B.P. is to require not less than 99 % of $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$. Talc, 1.0–6.6 %, was present in eight samples, an undesirable addition for an article used for infants' milk.

ANALYSIS. Talc should be removed as in the previous tablets, taking 1 gm. Evaporate the filtrate to dryness, ignite and weigh. Add 20 ml. N/2 HCl to ash, let stand, wash into flask and boil. Titrate with N/2 NaOH, using methyl red. In each case calculate to anhydrous citrate. The amount of moisture in the tablets should be determined.

PROSECUTION FOR BISMUTH DYSPEPSIA TABLETS. *London, Mansion House.* Deficient in bismuth carbonate 43 %, and in sodium carbonate 12 %, according to the label on the container. The retailers were prosecuted for selling, and the manufacturer for wilfully giving a false label. It was admitted that only half the proper amount of bismuth carbonate had been used owing to an accident. It was argued that the deficiency was not "wilful," and that the article was a proprietary one, outside the Act. Evidence was given that the article was not stamped, and another firm manufactured an article under the same description. The magistrate dismissed the case against the retailer, but fined the manufacturer £2 and 5 guineas costs, as there had been insufficient care in testing after manufacture (*B.F.J.*, 1931, 4).

COFFEE, SUGAR AND MILK TABLETS. TEA, SUGAR AND MILK TABLETS

Bradford. Each contained dried skimmed milk, and the coffee one, 1.5 % of chicory. For the defence it was stated that the presence of chicory was due to an accident, that they were compounded articles, and therefore any kind of "milk" could be supplied, as the Milk Regulations only applied to milk itself. The stipendiary held that offences had been committed, but, as it was a novel point, only fined the defendant 1s. in each case, with special costs (*Grocer*, 1917, June 16).

CHLORODYNE LOZENGES, JUJUBES

The 1885 B.P. introduced "Tincture of Chloroform and Morphia," which contained chloroform, morphine, prussic acid and

other ingredients, and was intended as a substitute for a proprietary "Chlorodyne," but that name was not used. Since then the name "Chlorodyne" has been extended to a number of, more or less, similar preparations.

PROSECUTION. *Nelson.* Chlorodyne lozenges. (Chloroform 1.85 % and none of the other ingredients of chlorodyne. Expert evidence was given that there was no standard for "chlorodyne," and that the articles sold as such differed in composition. The manufacturer said they were made from chloroform, sugar, gum acacia, gelatine, ether and water. The magistrates dismissed the case, as there was insufficient evidence to convict (*P.J.*, 1908, May 2; *B.F.J.*, 1908, 116).

Birkenhead. Wild cherry and chlorodyne jujubes. No chlorodyne. Fine £5 (*P.J.*, 1927, April 23).

BISMUTH LOZENGES

The 1885 B.P. required each lozenge to contain 2 grains of bismuth oxycarbonate.

PROSECUTIONS. *Skipton.* 1.54–1.56 grains of basic nitrate of bismuth per lozenge. Each lozenge was marked "B.P. Bismuth, 2 grains." The average total weight was 27 grains, which was practically correct. Another vendor's lozenges contained 1.45–1.52 grains per lozenge, and the average weight of them was 22 grains. Each vendor was fined £2 (*F. & S.*, 1894, Sept. 29).

Ingleton. Bismuth subnitrate 1.2 grains out of 25.4 grains, instead of 2 grains out of 27 grains. Fine 1s. (*F. & S.*, 1895, Dec. 21).

COMPOUND BISMUTH LOZENGES

The B.P. requires each lozenge shall contain 0.15 gm. each of bismuth oxycarbonate and heavy magnesium carbonate, and 0.3 gm. of calcium carbonate.

The average weight of eleven samples bought in Birmingham in 1930 was 1.60–1.87 gm. Nine samples showed a variation of 4–10 % from the correct amounts of the ingredients, but two samples traced to one maker had an average deficiency of about 15 % of each ingredient. The makers explained that an insufficient quantity of a mixture of the ingredients had accidentally been added to the rose basis. The eleven samples were free from talc, the ash insoluble in acid being 0–0.3 %.

Another sample had been prepared with a peppermint basis, and contained only 0.05 gm. of bismuth oxycarbonate, no calcium carbonate, and 0.258 of magnesium carbonate per lozenge; 3.5 % of talc was also present.

ANALYSIS. *Evans (C. & D., 1906, March 31)* gave the following method of analysis. Ignite gently with HNO_3 till all organic matter

is destroyed. Dissolve the ash in HCl and dilute, but not to precipitation, and precipitate with H_2S . Determine calcium and magnesium in the filtrate. Dissolve the precipitated bismuth sulphide, precipitate, and weigh as Bi_2O_3 .

TANNIC ACID LOZENGES

In 1912, nine samples examined in Birmingham contained 0.48–0.59 grain of tannic acid per lozenge, 0.5 grain being the proper quantity.

PROSECUTION. *Sunderland.* Starch 8 %, which was not mentioned in the B.P. formula. Evidence was given for the defence that starch was not incorporated in the basis, but used for dusting the lozenges so that they should not adhere to a sticky mass. A public analyst found 6 % of starch, and stated the usual amount of starch used was 2–5 % ; it was an addition, and not a substitution, and as long as 0.5 grain of tannic acid was present, the maker could use his discretion. The magistrates decided that the offence was not proved (*P.J.*, 1914, Oct. 31).

CHAPTER XXXVI

EXTERNAL REMEDIES

Belladonna plasters. Crushed linseed. Violet powder. Arsenical soap. Ointments :—Boric acid. Iodine. Mercury. Nitrate of mercury Calomel. White precipitate. Zinc. Sulphur. Cocaine. Carbolic. Resin. Liniments :—Ammonia. Turpentine. Turpentine and acetic acid. Soap. Methylated belladonna. Camphor, camphorated oil.

BELLADONNA PLASTERS

THE B.P. of 1898 required this plaster to contain 0.5 % of the alkaloids of belladonna root, but the plaster was found to be too strong, and the 1914 B.P. reduced the strength to one-half.

Methods for the determination of the alkaloids have been given by Bird (*S.P.A.*, 1899, **24**, 175 ; *P.J.*, 1899, ii., 147), Parker (*C. & D.*, 1899, ii., 331), and Smith (*Analyst*, 1898, **23**, 214) ; the latter gives analyses of eleven samples, some of which were of very deficient strength.

PROSECUTIONS. *Lambeth.* Only 50 % of B.P. strength. The defendant said the plaster was of the strength usually sold during the past twenty years, and that owing to its strength he would not sell the new plaster without a prescription. Fine £2 and costs (*F. & S.*, 1899, Feb. 4 ; *B.F.J.*, 1899, 58).

Liverpool. Belladonna alkaloids 0.05 %, being one-tenth of the B.P. strength. Fine £5 and costs (*C. & D.*, 1900, June 23 ; *B.F.J.*, 1900, 205).

CRUSHED LINSEED

The 1914 B.P. requires the presence of at least 30 % of oil, and not more than 5 % of ash, and no starch. The oil limit is low, as the lowest Birmingham sample free from starch contained 33.7 % of oil, another had 34 %, and fifteen others, some of which had a little starch, yielded 35–42 % of oil. The proportion of ash varied 3.4–4.2 %. A number of samples had been adulterated with barley and other starchy matter.

A little deficiency of oil may be due to leaving in a paper wrapper, but ground linseed cake containing little oil has been substituted. This is not suitable for poultices. To avoid misunderstanding, "Crushed linseed" should be asked for, and not "Linseed meal." Linseed meal for the uses of ships is required by the Board of Trade to contain powdered camphor as a preservative. Of the samples of "Linseed (crushed and meal)" analysed in England and Wales 1920–9, 2.2 % were adulterated.

If a *decoction* of linseed meal be tested for starch, care must be taken to add *excess* of iodine, as the reagent is taken up by the oil.

PROSECUTIONS. *Fife.* Linseed meal from which 19.6 % of oil had been abstracted. The sheriff dismissed the case, holding that there were two kinds of meal, and that the sale of linseed cake meal was justified, as there was no specification of what was wanted (*C. & D.*, 1898, Sept. 10 ; *B.F.J.*, 1899, 22).

Dundee. Bruised linseed meal deprived of not less than 26.3 % of its natural oil. The sheriff dismissed the complaint ; it was not asked for under the B.P. name and was not a drug (*B.F.J.*, 1901, 365).

Woking. Crushed linseed containing mites, traces of starch, and being deficient of 33 % of oil. Fine 10s. (*Grocer*, 1913, May 24).

Southampton. Linseed meal deficient in oil to the extent of 25.6 % of the standard minimum. The defence was that mustard-seed meal had been sold by mistake. Fine £5 (*Grocer*, 1922, Jan. 14).

London, Tower Bridge. Crushed linseed 50 % deficient in fat. It was stated that the article had been wrapped up for some time, and that the oil had been partly absorbed by the paper wrapping. Fine £2 (*Grocer*, 1926, Jan. 15).

VIOLET POWDER

In 1878 a wholesale chemist and drysalter was tried for the manslaughter of a baby by selling violet powder containing 38 % of arsenic, but was acquitted of criminal negligence. A number of other children died (*Analyst*, 1878, **3**, 280, 330). In that year forty-seven out of seventy-four samples examined by public analysts were reported adulterated (*Analyst*, 1879, **4**, 68). The editors of the *Analyst* asked about 1,000 practical pharmacists, "How do you make violet powder ?" Seventy-five % of the replies indicated starch and orris root, 20 % more also used magnesia, and in 5 % French chalk was given as a constituent (*Analyst*, 1879, **4**, 15). Dupré found that fuller's earth and starches were much more absorbent than crystallised calcium sulphate (*Analyst*, 1879, **4**, 60).

PROSECUTIONS. *London, Peckham.* Almost entirely sulphate of lime. Costs 12s. 6d. (*Analyst*, 1879, **4**, 14).

London, Tottenham. Calcium sulphate 53 %. Summons withdrawn, as the vendors had gone into liquidation (*P.J.*, 1904, Dec. 24).

ARSENICAL SOAP

PROSECUTIONS. *Brentford.* No arsenic. The manufacturer stated that he put $2\frac{1}{2}$ grains of arsenic in every 3 cwt. of soap. Fine £5 (*P.J.*, 1896 ; Dec. 11 ; *F. & S.*, 1896, Dec. 26).

Richmond. Soap free from arsenic 100 %. It was labelled "Arsenical Toilet Soap." The Government analysts reported that

if arsenic was present at all, it was in very minute quantity, and that the amount of soap received was not sufficient to detect $\frac{1}{100}$ grain per lb. For the defence it was argued that the soap was not a drug, and that proceedings should have been taken under the Merchandise Marks Act. The magistrates held, that while arsenical soap was a drug within the meaning of the Act, as the soap contained no arsenic it was not a drug, and dismissed the case. On appeal, *Houghton v. Taplin* (1897), the magistrates' decision that the soap was not a drug was confirmed (*P.J.*, 1897, Jan. 23; *F. & S.*, 1897, Feb. 13; *Analyst*, 1897, **22**, 167).

OINTMENTS

The departures from the authorised fatty basis, found in some purchased samples of ointments, suggest that the makers considered it a matter of indifference what basis was used, as long as the correct proportion of active ingredient was present. The use of an incorrect basis may diminish the activity of the drug by hindering its absorption.

In 1911 and 1913 Wild made Reports on the subject to the Therapeutic Committee of the British Medical Association (*Brit. Med. Jour.*, 1911, July 22; 1913, Oct. 18; *P.J.*, 1911, July 29; 1913, Nov. 1). His experiments showed that soft paraffin and paraffin ointment are hardly absorbed at all, but remain on the skin. About 15 % of lard and olive oil were absorbed after two minutes' rubbing, and 20 % of hydrous wool fat, provided it was not dried and had the proper amount of water present. He concluded that the glycerin esters of the higher fatty acids and the cholesterin fats are absorbed more readily than the hydrocarbons.

Papers on the analysis of ointments have been given by Elsdon, on phenol and salicylic ointments (*B.P. Conf.*, 1920, 476), and Evers and Elsdon (*S.P.A.*, 1922, **47**, 197). The latter paper gives methods of analysis for various ointments, and deals with the loss of volatile constituents during preparation, and with their refractive indices.

The determination of the B.R. of the bases obtained from an ointment by melting and filtration is sometimes useful. Hard paraffin may be tested for by heating the ointment in a small beaker on the water oven with B.P. H_2SO_4 , once or twice, and noticing if a more or less white cake is obtained on cooling.

Extensive alterations in the bases of ointments are suggested for the new B.P., and it is probable that ointments prepared according to the 1914 formula will be sold for some time after the issue of the new edition. Strictly speaking, such ointments should be marked 1914; but as the proposed changes will have little or no effect on the therapeutic action of the ointment, the writer

considers that it is allowable to pass unmarked ointments as genuine, until a reasonable time for them to be sold has elapsed.

BORIC ACID OINTMENT

This ointment is required by the B.P. to contain 10 % of boric acid.

BORIC ACID IN BORIC ACID OINTMENT (198 samples)

Boric acid, %	0.5-	8.5-	9.5-	10.5-	11.5-19.5	Total.
1909-31.	5	9	66	15	5	100

The extremes of 0.5 % and 19.5 % of boric acid show that in some cases the mixing was very carelessly done. That there is no real difficulty in mixing is shown by the fact that two-thirds of the samples were not more than 0.5 % from the 10 % required. The suggestion has been made that as boric acid is dried in centrifugal machines, the deficiency may in some cases be due to the use of damp boric acid. The samples were tested for hard paraffin by heating with B.P. H_2SO_3 in small beakers on the water oven. In each case a white cake was obtained on cooling, showing that paraffin ointment had been used. In a few instances the yellow variety had been incorrectly used.

Of the samples examined in England and Wales during 1907-13, 3.9 % were adulterated, and 5.1 % of those during 1920-30.

PROSECUTIONS. *London, Lambeth.* Boric acid 8.3 %, yellow paraffin ointment 91.7 %. The defendant's analyst found 9.4 %, and the Government analysts 8.7 %. They remarked that the ointment had been badly mixed, as parts of it varied from 7.7 % to 9.6 %. Fine 10s. (*P.J.*, 1901, Nov. 30, Dec. 14; *B.F.J.*, 1901, 396).

London, Lambeth. Boric acid 5.9 %, lard 94.1 %. Fine £3 and 17s. costs (*P.J.*, 1901, Nov. 16).

London, Shoreditch. Boric acid deficient 12 % of the correct amount. Paid costs (*P.J.*, 1925, May 2).

London, Tower Bridge. Boric acid 0.04 %, being a cheap soap jelly, and not an ointment at all. Fine £20. The wholesale dealer was subsequently fined £50 for adulteration (only 0.4 % of boric acid being present) and £20 for wilfully giving a false description (*B.F.J.*, 1930, 66, 76; *P.J.*, 1930, June 7, July 5).

IODINE OINTMENT

According to the B.P. of 1898, and that of 1914, this ointment when made should contain—iodine 4 %, potassium iodide 4 %, glycerin 12 %, and lard 80 %. No test for the finished product is given.

Pullen (*P.J.*, 1912, Nov. 16, and *Y.B.P.*, 1913, 355) has shown that there is combination between the iodine and the lard. During

the first day after making the free iodine fell from 4.0 % to 3.3 %, and four months later it was 2.92 %. A sample made with lard two years old fell to 3.15 % in one day, and 2.26 % in four months. At the end of that time the iodine in combination with the fat of the first sample was 0.95 % and the second had 1.62 %; there was therefore very little loss in the total amount. The colour of these ointments remained a rich chocolate. Six samples bought at various pharmacies contained 2.48–2.85 % of free iodine. The presence of potassium iodide is essential to limit this change.

The free iodine was determined by dissolving in chloroform, adding water and KI, and titrating with $\text{Na}_2\text{S}_2\text{O}_3$ solution. To determine the combined iodine the ointment was dissolved in chloroform, washed with a solution of $\text{Na}_2\text{S}_2\text{O}_3$, the residual fat distilled with strong H_2SO_4 , and the iodine weighed as AgI.

Experiments on the U.S.P. ointment, which is similar in composition, by Fried (*Y.B.P.*, 1915, 270) and Warren (*Y.B.P.*, 1918, 334) indicate that equilibrium is attained in that ointment when about 70 % of free iodine is present; see also Thompson and Snyder (*Y.B.P.*, 1917, 102).

PROSECUTION FOR IODINE OINTMENT. *Dublin.* Free iodine 2.39 %, instead of 4 %. The defendant's analyst found 2.12 % of free iodine, and 2.03 % of iodine combined with lard. The summons was withdrawn (*P.J.*, 1912, May 11).

MERCURY OINTMENT

The London Pharmacopœia prescribed two mercury ointments—a strong and a diluted, one-third the strength. In later Pharmacopœias there has been only one mercury ointment; that of 1885 and 1898 contained 48.5 % of mercury, and that of 1914, 30 %.

The B.P. ointment, owing to its strength, is rarely sold unless by medical prescription, and a weaker form, sometimes containing about 16 % of mercury, is sold under various names: troopers' ointment, blue ointment, blue butter. The latter is used to destroy crab-lice, and for sheep and other animals. A distinction has been made between "mercury ointment," the B.P. article, and "mercurial ointment," a weaker preparation. The B.P. Codex prescribes a diluted "Mercurial ointment" having 10 % of mercury.

Of the samples of mercurial ointment analysed in England and Wales during 1900, 1902, 29.7 % were adulterated, and 17.2 % of those examined 1921–30.

ANALYST. Treatment of mercury ointment with ether may remove oleate of mercury with the fat, so that for the determination of the total mercury concentrated nitric acid must be used. Methods of determination have been given by Rupp (*Y.B.P.*, 1907, 103),

Crewe (*B.P. Conf.*, 1908, 473), and Roberts (*B.P. Conf.*, 1924, 719).

PROSECUTIONS. *Skipton.* Mercury 12.5 %, instead of 48.5 %, to the prejudice of the purchaser. The box was labelled "Mercurial Ointment (Poison)." As it was a "compounded article," it was contended for the defence that proceedings should have been taken under sect. 7 of the 1875 Act, and not under sect. 6. This objection was overruled. Pharmacists of long experience gave evidence that, owing to the danger to the public in using the B.P. strength, it was customary to supply the reduced form, except on medical prescriptions. The authority of the B.P. as governing the composition of articles sold under the English name was also challenged. The justices found that the commodity sold was not of the nature, substance and quality of the article demanded by the purchaser, and held that the vendor was bound to supply the commodity according to the formulary prescribed in the B.P., whether expressly asked to do so or not. Fine £3. An appeal to the Court of the King's Bench followed, *Dickens v. Randerson* (1901). The judgment of the Court stated that the appellant when asked for mercury ointment should have sold the B.P. drug, or have explained that he was selling a diluted drug, and have so named it. No attempt had been made to prove any other commercial standard than that of the B.P., and the suggestion that there were two commercial standards of wide difference was unreasonable. If a drug found in the B.P. be asked for, that drug must be supplied, and if it be not sold with the ingredients and in the proportions prescribed by the B.P., there is at least *prima facie* evidence that what was being sold was not of the nature, substance and quality demanded. It was also decided that a "compounded drug" of sect. 7 was also a "drug" under sect. 6. The conviction was affirmed (*Analyst*, 1901, 26, 54; *B.F.J.*, 1900, 204; 1901, 88; *C. & D.*, 1900, July).

Edmonton. Mercury 31.58 % and prepared with lanoline. The magistrate said that if the defendant had marked the article "Not British Pharmacopœia," it would have been sufficient notice, and fined defendant 5s. (*C. & D.*, 1900, Oct.; *B.F.J.*, 1900, 296).

London, Mansion House. Mercury deficient 70 %. There was a written order "Ung. Hydrarg. ℥iii." Nominal penalty of 2s. 6d. (*B.F.J.*, 1902, 45).

Liverpool. Devoid of mercury. Fine £2 and costs (*C. & D.*, 1902, Aug. 2; *B.F.J.*, 1902, 185).

Liverpool. Mercury deficient 42.3 %. The box was plainly marked "diluted," and the purchaser was aware of the fact. Case dismissed (*C. & D.*, 1902, Sept. 6).

Longton. Eight defendants were prosecuted for deficiencies in mercury varying from 21 % to 82 %. In some cases the article was

marked "diluted," but wrapped in an opaque wrapper. The stipendiary decided that in each case an offence had been committed, the drug asked for not having been supplied. In reference to the labelled samples, wrapped in opaque paper, he found that the purchaser had not, nor could he reasonably be expected to have had, a knowledge of what (if anything) was on the label. Each defendant was fined £2 (*C. & D.*, 1902, Dec. 27; 1903, Jan. 10, 17; *B.F.J.*, 1903, 19).

Portsmouth. Mercury deficient 62 %. The assistant stated he had sold "Blue ointment." Fine £5 (*P.J.*, 1920, Aug. 28).

NITRATE OF MERCURY OINTMENT

PROSECUTION. *London, Mansion House.* Deficient in mercurial content, calculated as metallic mercury, at least 66 %. Fine £10 (*B.F.J.*, 1931, 35).

CALOMEL OINTMENT

The strength of this ointment has been varied by each Pharmacopœia. The 1885 edition specified 15.5 %, the 1898, 10 %, and the 1914, 20 %. A 33 % strength is also in use.

PROSECUTIONS. *London, Bow Street.* Calomel 20.6 %, and 10 %, respectively, instead of the 33 % strength asked for. Ordered to pay 3 and 5 guineas costs, respectively (*P.J.*, 1921, Feb. 5; *B.F.J.*, 1921, 15).

London, Bow Street. Mercurous chloride 11.5 %, instead of 20 %. Fine £7 (*P.J.*, 1925, Oct. 31; *Analyst*, 1925, 50, 611).

London, Mansion House. Ammoniated mercury 4.6 % and no mercurous chloride. It was stated that a mistake had been made. Fine £5 (*B.F.J.*, 1927, 114).

London, Bow Street. Calomel 9.8 %, instead of 20 %. The Government analysts found 10.5 %, and the defendant's analyst found 12 % or more. It was suggested that the ointment had been allowed to get hot, and that the calomel had partly separated. Analysis of the rest of the consignment showed that part of it was over strength. Fine £5 (*P.J.*, 1929, Feb. 2, March 9).

WHITE PRECIPITATE OINTMENT

This preparation is required by the B.P. to contain 5 % of ammoniated mercury, which itself must contain not less than 94.5 % of mercuric-ammonium chloride (NH_2HgCl). Previous to 1914 the ointment contained 10 %.

This ointment is a scheduled poison, and is legally required to be labelled "White Precipitate Ointment, Poison," with the name and address of the seller. A number of Birmingham samples were unlabelled or bore incomplete labels. The ointment may only be

sold by a registered pharmacist. Eight samples bought from unqualified vendors contained no white precipitate; one was boric acid ointment, another was zinc carbonate ointment, and six were zinc ointment containing 6.9–15.9 % of zinc oxide. Substitutes are also sold with names resembling “precipitate.”

STRENGTH OF WHITE PRECIPITATE OINTMENT, 1919–29
(fifty-six samples)

Mercuric-ammonium chloride, %	2.2–3.3	4.0–	4.5–	5.0–	5.5–7.5	Total.
Percentage of samples	5	15	40	31	9	100

The proportion of samples more than 10 % different from the correct proportion is too large. Some of the errors are probably due to gross carelessness, but white precipitate is a heavy powder, which will require careful mixing, and which may possibly separate in warm weather, or if heated during preparation. In two instances paraffin ointment had been used instead of benzoated lard.

ANALYSIS. The following method of determination is based on that of Elsdon (*B.P. Conf.*, 1911, 447). Weigh about 2 gm. of the well-mixed ointment in a small funnel with short stem. Place funnel in neck of separator, and put in water oven till melted. Wash funnel with 20 ml. 3E.HCl, then with 15 ml. petrol, and finally with 10 ml. boiling 3E.HCl. Shake and run out the acid liquid after separation. Wash with hot water, followed by two treatments with 10 ml. boiling 3E.HCl. Keep separate two further treatments, which are each tested to see that extraction is complete. Cool main liquid, add 30 ml. 3E.NaHO, dilute to about 200 ml. and precipitate with H_2S . The residual fat solution should give no black specks on addition of ammonium sulphide. The ointment should not yield more than 0.1 % of ash.

Allport has recently advocated the use of a solvent composed of—glacial acetic acid 50 v/v, benzene 45 v/v, and 90 % alcohol 5 v/v (*Q.J.P.*, 1928, 25).

PROSECUTIONS. *Birmingham.* Zinc carbonate 9.5 %. The vendor was unqualified, and on promising to give up the business, was ordered to pay costs only (1902 Report).

Birmingham. Zinc oxide 6.9 %. Fine £2 (*B.F.J.*, 1909, 154).

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ZINC OINTMENT

This ointment should contain 15 % of finely sifted oxide of zinc, and if the B.P. directions “stir the mixture constantly till cold” be not followed, the oxide may settle during cooling, and the lower part be stronger than the upper. This is the probable explanation of samples containing 11.8 % and 23.7 % of zinc oxide. There is the possibility of the accidental substitution of other white ointments, such as boric acid, or lead carbonate, but the ash in such cases will

not be yellow while hot and white when cold, and its proportion will also be much less.

The B.P. for 1914 required the use of benzoated lard, but in the new edition the basis is to be altered.

ASH IN ZINC OINTMENT (166 samples)

Ash, %	.	.	11.8-	14.5-	15.5-	16.5-	18.5-	20.5-23.7	Total.
1913-20	.	.	9	34	26	16	9	6	100
1921-8	.	.	17	59	20	3	1	0	100

The latter period is distinctly better than the former one, as in it 59 % of the samples were within 0.5 % of the correct proportion, as against 34 % in the first period. Some, at any rate, of the improvement may be attributed to the number of cautions given.

In one case it was noticed that as the cardboard box containing the ointment became oily, the percentage of ash increased from 12.8 to 14.4 %.

Of the samples examined in England and Wales, 4.7 % were condemned during 1920-30.

PROSECUTIONS. *Blaenavon.* Oxide of zinc 8 %, common lard 92 %. The manufacturers presumed the workmen had failed to stir the ointment during cooling. Fine £2 (*B.F.J.*, 1901, 29).

Pocklington. Zinc oxide and benzoated lard entirely absent. The defence was that boracic ointment had been served by mistake. Fine 2s. 6d. (*P.J.*, 1914, Dec. 12).

Bradford. Dry white lead 10 %, lard 90 %. The Medical Officer of Health gave evidence of the danger due to white lead being readily absorbed by the skin. The defendant, who admitted that a boy had supplied the substitute as there was no zinc ointment in stock, was fined £5 (*P.J.*, 1915, April 24; *B.F.J.*, 1915, 100).

Bradford. Zinc oxide 11.4 %. Defendant pleaded that there was no fraud, as the benzoated lard was more costly than the zinc oxide which it replaced. The oxide used was said to have been gritty, which made it more liable to sink in the medium. Fine £2 (*P.J.*, 1916, April 22; *B.F.J.*, 1916, 389).

SULPHUR OINTMENT

The 1898 B.P. reduced the proportion of sublimed sulphur in this ointment from 20 % to 10 %. Ten of the eighteen Birmingham samples examined in 1917 and 1924 were within the limits 9.5-10.5 %; five contained 8.9-9.3 %, and three 10.6-10.7 %. Benzoated lard, the basis of the 1914 B.P., had been used in seventeen samples, the B.-R. 40° of the filtered fats being 50-56. The fat of one sample had a refraction of 67, and it only required 7.2 % of KHO for its saponification.

Two samples from one vendor had about 19 % of sulphur; the

fat had a refraction of about 80 and a Wijs iodine absorption of about 15 %.

ANALYSIS. Heat about $\frac{1}{2}$ gm. with 10 ml. HNO_3 in 250 ml. conical flask on asbestos plate and keep nearly boiling. Add a few fragments of KClO_3 and KBr , and repeat treatment once or twice if the oxidation of the sulphur appears incomplete. Add 100 ml. hot water, filter and wash after standing half an hour on the water bath. If any globules of sulphur are filtered out, they should be weighed. Neutralise the filtrate with AmHO , acidify slightly with HCl , and precipitate with BaCl_2 .

Henville (*S.P.A.*, 1930, **55**, 385) has pointed out the error that would arise if the incorrect assumption be made that sulphur is insoluble in petroleum spirit.

PROSECUTION. *Birmingham.* Sulphur 19 %, paraffin basis 81 %. The defendant stated that his customers preferred the stronger preparation. He sold the ointment at a lower price than other chemists, but did not think it was illegitimate competition. Ordered to pay 5s. costs under the Probation of Offenders Act (*P.J.*, 1924, July 26).

COCAINE OINTMENT

The 1898 and the 1914 B.P. require this ointment to contain 4 % of cocaine. Cocaine hydrochloride contains 89.3 % of cocaine.

PROSECUTION. *Bournemouth.* Cocaine hydrochloride 3.92 %, benzoated lard and oleic acid 96.08 %. The order for the article was given by a prescription ordering "B.P. cocaine ointment." An objection to the certificate was overruled by the Bench and the vendor ordered to pay 41s. costs (*P.J.*, 1904, July 30).

CARBOLIC OINTMENT

Although the B.P. requires the ingredients to contain 3 % of carbolic acid, there is some loss in making it. For the analysis, see Elsdon (*B.P. Conf.*, 1920, 476).

PROSECUTION. *London, Marylebone.* Deficient in phenol, containing only 2 %, and adulterated with 21.6 % zinc oxide, 20 % starch, and 6.8 % sulphur. The vendor, a herbalist, was fined £2 (*P.J.*, 1931, Jan. 10).

RESIN OINTMENT

PROSECUTION. *Bradford.* Resin 5 %, neutral oil 3.5 %, foreign matter, chiefly vaseline, 91.5 %. Lard and beeswax were absent. Evidence was given that the B.P. ointment was much superior and more than twice as costly. The defendant said that he and his father had been selling the article as "Resin healing ointment" for fifty years. Fine 10s. (*P.J.*, 1914, Dec. 12).

LINIMENT OF AMMONIA

According to the B.P., this liniment should be made by shaking together 1 volume each of solution of ammonia and almond oil, and 2 volumes of olive oil. In 1908 the Public Analyst for Camberwell reported that no less than fourteen of the twenty-three samples analysed failed to attain the B.P. standard. Two of them were deficient in oil, eight in ammonia, and four in both constituents. Two of the latter contained 5.2 % and 28.3 % of oil of turpentine. The deficiencies varied from 11–42 %. Three vendors were fined £1–£2.

PROSECUTIONS. *London, Lambeth.* Almond and olive oils 51.3 % instead of 74 %. Fine £1 and 17s. 6d. costs (*B.F.J.*, 1908, 211).

Rochester. Deficiency of oils 46.4 %, and contained 36.5 % turpentine. It was labelled “Liniment” only. The vendor was fined £1, the Bench considering it was a technical offence (*P.J.*, 1908, May 6).

LINIMENT OF TURPENTINE

PROSECUTIONS. *North London.* Non-volatile matter 266 parts by weight, rectified oil of turpentine 300 fluid parts, distilled water, and other volatile matter, up to 1,000 fluid parts, being deficient of 53 % of the oil of turpentine required by the B.P. The purchaser was given a choice of three liniments and accepted the white one ; it was labelled “Turpentine liniment, L.I.P.,” and had been prepared according to the London Insurance Pharmacopœia. The vendor was ordered to pay costs, as the purchaser did not get the actual article he required. On appeal to Quarter Sessions, the conviction was quashed as the Court believed the vendor delivered the article he thought was required (*P.J.*, 1928, Jan. 14 ; *B.F.J.*, 1928, 15 ; *Analyst*, 1928, **53**, 220, 282).

London, Old Street. Soft soap 8.5 gm. %, camphor and turpentine 35 ml. %, water 56.5 ml. % ; the combined camphor and turpentine were deficient to the extent of 50 %. The certificate was objected to, because it did not distinguish between the camphor and the turpentine, either or both of which might have been deficient. The case was dismissed (*P.J.*, 1930, Oct. 25 ; *Analyst*, 1930, **55**, 752).

London, Old Street. Deficient in rectified oil of turpentine 55 %. For the defence it was stated that the B.P. liniment was very strong and rarely asked for except on a doctor's prescription ; about twelve times as much of the N.H.I. white liniment was sold. The bottle was incorrectly labelled. Fine £3 (*P.J.* and *C. & D.*, 1930, Dec. 6).

LINIMENT OF TURPENTINE AND ACETIC ACID

PROSECUTION. *London, Old Street.* Glacial acetic acid 15 % deficient, extraneous water 36 %, and almost entirely deficient in

liniment of camphor. The defence stated that a commonly used "turpentine liniment" had been supplied. The vendor was ordered to pay costs, the magistrate considering that a mistake had been made, and thought it unfortunate that the compound liniment had not a special name (*P.J.*, 1927, Jan. 8).

SOAP LINIMENT

PROSECUTIONS. *London, Clerkenwell.* Made with methylated spirit instead of rectified spirit. Fine £20 and 10 guineas costs (*C. & D.*, 1901, Feb. 1).

North London. Methylated spirit present. Case dismissed, as the Government analysts failed to find it (*P.J.*, 1902, Feb. 15).

London, Clerkenwell. Methylated alcohol 50 %. The magistrate convicted the defendant company, after refusing to hear any evidence as to the existence of a commercial standard other than that of the B.P. On appeal, *Boots, Ltd. v. Cowling* (1903), he was directed to hear such evidence. After a hearing lasting several days, the magistrate decided there was no such commercial standard, and fined the defendant company £5 and 65 guineas costs (*C. & D.*, 1903, March 28, May 16, June 13, July 4; *B.F.J.*, 1903, 85, 139, 163).

Bradford. Camphor absent, and soft soap $\frac{3}{4}$ % instead of $9\frac{2}{3}$ %. Fine 10s. (*P.J.*, 1911, June 10).

METHYLATED BELLADONNA LINIMENT

PROSECUTION. *London, Highgate.* Alkaloids of belladonna 40 % deficient, 0.22 w/v being present instead of 0.375 w/v. It was compounded with methylated spirit. Case dismissed, as the analyst had treated it as if it were a B.P. article, whereas it was not sold as such (*C. & D.*, 1901, Dec.).

LINIMENT OF CAMPHOR. CAMPHORATED OIL

Since 1898 this preparation has been a 20 % solution of flowers of camphor in olive oil. The 1914 B.P. directs "Dissolve in a closed vessel." If the oil be first placed in the bottle, and the flowers which have been rubbed through a No. 20 sieve be added, solution will take place in a short time. If the olive oil be congealed, it may be warmed *before* the addition of the camphor. Many prosecutions have resulted through the drug being heated during preparation in an open dish; one-third, or more, of the camphor may be lost.

During war-time, 1918-19, permission was officially given to use other oils, and a mineral oil was recommended. Arachis or sesame oil may be used in India and other countries.

In a prosecution, camphorated oil was described by the defence as "an exceedingly volatile liquid," and similar explanations for the

deficiency of camphor have been given. Experiments by various people do not support this theory. The writer found that a sample kept in a corked bottle over two years only lost 1.0 % of camphor, and one left in an *open beaker* three days during hot weather only 0.3 %. Clover kept part of a sample of camphorated oil in a closed Winchester quart, and part in a frequently opened capped shop bottle during hot weather in August and September ; the difference between the two was only 0.2 % (*P.J.*, 1900, Nov.). A sample kept by Cripps in sunlight during five summer weeks lost 0.37 % of camphor (*P.J.*, 1904, Sept. 10). A winter experiment was made by Bevan, when a sample kept in an open medicine bottle thirty-three days lost 0.12 % (*P.J.*, 1907, July 20). Chapman, with $\frac{1}{2}$ oz. camphorated oil in a 16-oz. bottle, imitated the operation of pouring two or three times a day for eight days, and found 0.24 % loss (*P.J.*, 1907, July 20). In each of these experiments at least 20 % of camphor was present.

Camphorated oil was first bought in Birmingham for analysis in 1899, and in seven years twenty-five of the 103 samples examined were adulterated. Some were only deficient in camphor, but others had also cheaper oils present. A few of them, in later years, had little rotation, owing to the presence of synthetic camphor. During the years 1920-31, samples from three vendors contained only 14.5-18.1 % of camphor, and the others :—

CAMPHOR IN CAMPHORATED OIL (Two methods)

Percentage of camphor .	19.3-	19.8-	20.2-	20.6-21.0	21.5-22.4	Total.
Percentage of samples :						
Polarisation method .	20	30	17	20	13	100
Loss on heating method	24	27	18	20	11	100

It is evident that there is no difficulty in supplying camphorated oil containing at least 19.3 %, which corresponds with a deficiency of $3\frac{1}{2}$ % of the standard quantity. Some of the higher percentages may be due to extra camphor being used to compensate for possible volatilisation.

SP. GR. OF CAMPHORATED OIL, 1920-31 (Fifty-five samples)

Sp. gr.925	.926	.927	.928	.929	.931	Total.
Percentage of samples .	4	16	51	20	7	2	100

After correcting the sp. gr. according to the percentages of camphor present, the sp. gr. of the oils used in making 89 % of the camphorated oil varied from 0.918-0.920. The B.-R. 40° of 95 % of the samples of camphorated oil were 53-55. These figures give no indication of the presence of foreign oil.

Of the samples of camphorated oil examined in England and Wales during 1900-13, 8.1 % were adulterated, and 6.5 % of those examined 1919-30.

ANALYSIS. *Polarisation.* It has been stated previously (p. 487) that the specific rotation of natural camphor dissolved in oil is 54° , which corresponds to $+108^\circ$ in a 200-mm. tube. The following formula may be used, in the absence of synthetic camphor :—

$$\text{Camphor \%} = \frac{100 (L - o)}{(108 - o) S}$$

where "L" is the rotation of the liniment with a sodium flame in a 200-mm. tube ; "o" = the rotation of the oil alone in 200-mm. tube, and "S" = sp. gr. of the liniment. With olive oil the divisor is practically 100, and the value of "o" is $+0.3^\circ$. The formula then simplifies to: camphor % = $L - 0.3$. For samples of arachis, cottonseed and colza oils, the value of "o" was found to be negligible, two samples of sesame gave $+0.5^\circ$ and $+0.9^\circ$, and two samples of mineral oil gave $+1.0^\circ$. When mineral oil is present its lighter sp. gr. must be taken into account (see Liverseege, *C. & D.*, 1899, Jan. 28 ; 1901, Jan. 26 ; also Leonard and Smith, *S.P.A.*, 1900, **25**, 202 ; von Freidrichs, *Analyst*, 1918, **43**, 409).

Loss on Heating. The volatilisation of camphor from the liniment when heated in the water oven is slow, and was not complete in ten hours. but on the water bath, where the heavy camphor vapour can dissipate, the loss was complete in two hours. About 2 gm. should be taken in a flat-bottom metal dish, about 3 inches in diameter. The amount of loss will depend, not only on the amount of camphor, but also on any alteration of the oil itself on heating. One sample of olive oil was unaltered on heating, another gained 0.4 %, and an old sample lost 3.9 %. For ordinary olive oil samples, it is better to take the loss on heating as being camphor. In the analytical table given above it will be seen that there is a good agreement between the two methods of determination.

With other oils there was a gain on heating : arachis, 0.2 % and 0.6 % ; sesame, 0.4 % and 0.5 % ; cottonseed, 1.1 % and 1.3 %. On the other hand, mineral oil steadily loses, a sample made with it lost 48 %, while polarisation indicated 22 % (see Liverseege, *opus cit.* ; Leonard and Smith, *S.P.A.*, 1898, **23**, 281).

Synthetic Camphor. A sample of liniment prepared with synthetic camphor lost 17.7 % on heating, but 200 mm. only rotated 0.6° . Richardson and Walton have investigated the detection of camphor substitutes (*S.P.A.*, 1908, **33**, 463).

Specific Gravity. Experiments with olive oil indicated that 1 % of camphor increased the sp. gr. of the oil by 0.0004, a factor which may be used to calculate the sp. gr. of the oil itself. Other oils gave factors varying from 0.00035 (arachis) to 0.00058 (mineral).

Valenta Test. This value is lowered by about 2° for 1 % of camphor.

Butyro-refractometer Readings. The solution of about 20 % of

camphor in olive or arachis oils *increased* the reading by 0.3 ; with sesame and colza oils, the reading was *decreased* by 1.3, while mineral oil gave a larger decrease (Liverseege, *C. & D.*, 1901, Aug. 31).

Iodine Value, Saponification Value. Camphor may be taken as having no effect on these figures except as a diluent.

Foreign Oils. The writer (*opus cit.*) has shown that, by the methods given above, the constants of the oils used may be, with fair accuracy, calculated from the analyses of the liniments.

PROSECUTION FOR CAMPHOR LINIMENT. *Birmingham.* Camphor 10 %. It was ordered on a prescription. Fine £1 (*F. & S.*, 1897, Aug. 28).

PROSECUTIONS FOR CAMPHORATED OIL. *Ripon.* Camphor 14 %, sesame oil 86 %. Fine £2 (*F. & S.*, 1899, April 29).

Birmingham. Two chemists were each fined £1 for deficiencies of 13 % and 76 % of camphor, another £2 for 34 % deficiency, and another £2 for the presence of at least 50 % of foreign oil, with 13 % of camphor deficient (*F. & S.*, 1899, Dec. 16).

Warminster. Camphor 8 %, being less than half the proper quantity, and not of the nature, substance and quality of the article demanded. The magistrates dismissed the case, holding that camphorated oil was a "compounded drug" and that the summons had been taken out under the wrong section. This decision was reversed by the High Court on appeal, *Beardsley v. Walton* (1900, *B.F.J.*, 76).

Lambeth. Camphor 4.8 %, mineral oil 95.2 %. The bottles were labelled "Campholeum, formerly known to the Public as Camphorated Oil." Objection was made to the contents of three bottles being mixed and divided, but the method was held to be correct. Fine £2 (*B.F.J.*, 1900, 141, 173).

Luton. Camphor 17.45 %. The purchaser did not open any of the six bottles bought, or mix or divide the contents, but put two bottles in each of three bags. There was no evidence as to the bottles being identical in character, appearance or labelling. The magistrates were not satisfied that the two bottles reported on were identical in nature and substance with the other two sets of bottles, and therefore that the division was improper, and dismissed the case. This decision was upheld on appeal, *Mason v. Cowdary* (1900). The inspector had bought six articles and did not divide any of them, but divided the six into three parts (*B.F.J.*, 1900, 25, 162).

Birmingham. Camphor 16.7 %. Fine £20 ; an appeal, of which notice was given, was afterwards withdrawn (*C. & D.*, 1901, Jan. 26 ; *B.F.J.*, 1901, 101).

Birmingham. Camphor 8.0 %. For the defence it was pleaded that the proper proportions had been used, but that the preparation might have been allowed to boil. Fine £5 (*P.J.*, 1901, July 24 ; *B.F.J.*, 1901, 242).

Sheffield. Camphor 5 %, fish oil 95 %. The article was stated to have been prepared from camphor 10 %, turpentine 10 % and colza oil 80 %, and to be "Camphor Embrocation." Fine £1 (*B.F.J.*, 1901, 300).

London, South-Western. Deficiency in camphor 18.6 %. Evidence was given for the defence that, as the article was liable to evaporation and rancidity, the certificate should have reported if there was any decomposition. The magistrate held that volatilisation-decomposition did not come within the meaning of the note to the certificate. It was also argued that as the B.P. gave no "test," there was no standard. The magistrate held that, as the two simple ingredients were merely blended, a "test" was superfluous. Camphor was an obvious constituent, and not like acetic acid in vinegar of squill. Fine £1 (*P.J.*, 1904, July 23, Oct. 29, Nov. 12; *B.F.J.*, 1904, 260).

Liverpool. Mineral oil and without camphor. Fine £5 (*P.J.*, 1907, March 9).

London, Old Street. Camphor 5 %, paraffin oil 95 %. Fine £10 (*P.J.*, 1908, April 4; *B.F.J.*, 1908, 81).

Pocklington. Lard oil 30 %, and deficient of 51 % camphor. Fine 10s. (*P.J.*, 1910, Dec. 10).

Stourbridge. Artificial camphor. Fine £5 (*P.J.*, 1912, March 30; *B.F.J.*, 1912, 75).

Breaston. Mineral oil, and 44 % of camphor deficient. The bottles were labelled "Camphor and Oil," Case dismissed. (*P.J.*, 1913, Aug. 23).

Bristol. Camphor 12½ % deficient. The oil was stated by the defence to be an exceedingly volatile liquid, and that in the winter months, re-heating was necessary. Fine £15 (*B.F.J.*, 1930, 26).

PROSECUTION FOR CAMPHOR AND MUSTARD OIL.

Sunderland. Camphor 3.35 %, mineral oil 67.42 %, saponifiable oil (which might or might not contain mustard oil) 29.23 %. The objection was to the mineral oil. Paid £5 (*B.F.J.*, 1922, 55; *Grocer*, 1922, May 27).

CHAPTER XXXVII

MISCELLANEOUS DRUGS

Glycerin. Glycerin of borax. Glycerin and lime cream. Liquid extract of cascara sagrada. Compound decoction of aloes. Hypodermic injection of morphia. Solution of ammonium acetate. Solution of iodine.

GLYCERIN

A RANGE of sp. gr. 1.260–1.265 is suggested for the new B.P. corresponding to 98–100 % purity, and Birmingham samples did not differ seriously from that figure.

SP. GR. OF GLYCERIN (Sixty-nine samples)

Sp. gr.	1.250–	1.255–	1.260–	1.265	Total.
Percentage of samples	9	23	59	9	100

None of the forty-two samples examined since 1914 exceeded the B.P. limit of 4 per million of arsenic, and only one-sixth of them had that amount. Two vendors were cautioned in 1915 because of the presence of lead, 16 and 7 parts per million, respectively, being present: thirteen examined 1924–9 were free from lead; two others had 4 and 2 parts per million, respectively. Of the 121 samples examined 1888–1929, two, from one vendor, were adulterated with 12 % of water, and two with glucose syrup; the latter were taken from penny bottles.

Of the samples examined in England and Wales 1897–1905, 5.7 % were adulterated; during 1906–13 the proportion of adulteration fell to 2.6 %, and 1920–30 to 1.0 %.

ANALYSIS. New sp. gr. tables have been given by Bosart and Snoddy (*Analyst*, 1927, **52**, 434). The coefficient for 1° C. is 0.0006. Hehner devised a bichromate method for the determination (*S.P.A.*, 1887, **12**, 44). International standard methods (*Analyst*, 1911, **36**, 314) include a modification of it, and also the acetin method. The determination of glycerin in spirituous galenical preparations has been described by Naylor and Chappel (*B.P. Conf.*, 1909, 260; *Analyst*, 1909, **34**, 440), the determination in egg yolk by Cockburn and Love (*S.P.A.*, 1927, **52**, 143), and in cream by Lerrigo (*S.P.A.*, 1928, **53**, 335). Chapman has studied qualitative tests for glycerin (*S.P.A.*, 1926, **51**, 384). Hehner has described experiments on the non-volatility of glycerin with aqueous vapour (*S.P.A.*, 1887, **12**, 65).

One of the samples adulterated with glucose syrup had $[\alpha]_D$ 32, and K of 11.5; if the corresponding figures for the adulterant had

been 112 and 35, respectively, 28 % or 23 % of glucose syrup would be indicated. The syrup, however, would require thinning with water, the latter increasing the amount of adulteration. The solids were determined by weighing about 5 gm. in a tared beaker having a glass rod, and dissolving the glycerin by treatment with a mixture of 2 parts of absolute alcohol with 1 of chloroform. The residual solids were weighed after drying in the water oven. The solids in the sample amounted to 23·2 %, or, as the sp. gr. of the sample was 1·274, 29·6 w/v. According to Allen, a 10 w/v solution of starch glucose has sp. gr. of 1·0384, and from this figure it may be calculated that 1 gm. of the solids has, in solution, a volume of 0·62 ml. Assuming that this value is true in the presence of glycerin, and that there is no contraction on mixing, the following calculation was made :—

Sample of "glycerin," sp. gr. 1·274	. 100·0 ml.	weight	127·4 gm.
Solids, in 100 ml.	. 18·2 "	"	29·6 "
			<hr/>
Difference	. 81·8 "	"	97·8 "
Equal to	. 1·0 "	"	1·2 "

According to glycerin tables, sp. gr. 1·2 corresponds to 75 % of anhydrous glycerin.

Anhydrous glycerin = (100 — 23·2) 0·75	. .	57·7 g.m
Solids	. 23·2	
Water (by difference)	. 19·3	
		<hr/>
Dilute glucose syrup	. 42·5	42·5 "
		<hr/>
		100·0

The sample was certified as containing "about 40 % dilute glucose syrup."

PROSECUTIONS. *Leeds.* White arsenic 2 grains per lb. The purchase was a 1-lb. bottle labelled "Warranted pure." Fine £3 (*F. & S.*, 1894, July 14).

Birmingham. Dilute glucose syrup about 40 %. Fine £5. Another vendor paid the same fine for about 45 % (*P.J.*, 1897, Feb. 27; *Analyst*, 1897, **22**, 112).

Castle Eden. Added water 14 %. The article was sold in penny bottles, labelled "Re-distilled." Fine £1 (*F. & S.*, 1897, March 20).

Southwark. Extraneous water 7 %. It was said to have been in stock two years, and evidence was given that glycerin exposed to the air absorbed 2·7 % of water in one night, and 10·6 % in eight days. Paid costs (*C. & D.*, 1903, Feb. 28; *B.F.J.*, 1903, 68).

Bradford. Arsenic 11 parts per million, Fine £1 (*P.J.*, 1905, July 8; *B.F.J.*, 1905, 158).

London, Thames. Sugar 29 %. The sample was taken from penny bottles supplied by a wholesale dealer, who was fined £10 (*P.J.*, 1913, June 21 ; *B.F.J.*, 1913, 110).

GLYCERIN AND LIME CREAM

PROSECUTION. *Brentford.* The Public Analyst found no glycerin, and the Government analysts " $\frac{1}{2}$ fl. drachm in 8-oz. bottle." The Bench dismissed the case without costs, and considered the discrepancy to be due to separation of the glycerin (*C. & D.*, 1897, May 15).

GLYCERIN OF BORAX

According to the 1898 and 1914 editions of the B.P., this preparation should contain 11.7 % of borax, and 83.7 % of B.P. glycerin, which contains about 2 % of water.

Its sp. gr. should be about 1.28 and the B.-R. 12.5° about 56. The twenty-two Birmingham samples examined early in 1914 showed considerable variations. Fifteen of them approximated to the B.P. composition, having 10.9–13.0 % of borax, and 0.9–2.9 % of water, about 2 % of which being due to the water in the glycerin. Four samples were weak in borax, 3.2–9.6 % being present ; the water in these samples varied from 3.7 % to 10.0 %. Three others were too strong in borax, 15.3–18.4 % being present, with 19.5–26.0 % of water. The amounts of water in the latter approximated to that of the 1885 B.P., but the proportions of borax were decidedly higher than the 12.4 % required by that edition. The unsatisfactory samples were obtained from six vendors, of whom three were prosecuted and two cautioned.

The borax was determined by weighing about 4 gm., adding a little water, neutralising to methyl orange, adding phenol phthalein, and titrating with N/2 NaHO. The result was calculated both to $\text{Na}_2\text{B}_4\text{O}_7$, and also to B.P. borax.

For the determination of glycerin, the refraction was taken with the butyro-refractometer at 12.5° C. By the use of Skalweitz's table, the following formula has been calculated, Z being the refractometer reading. $\text{Glycerin \%} = 80 + 4.7 (Z - 30)$. On the assumption that the anhydrous borax has no effect on the refraction, the above calculation will give the percentage of glycerin in the mixture of glycerin and water present—a figure which will require reduction according to the percentage of anhydrous borax present. For example, a sample prepared in the laboratory, containing 6.1 % of anhydrous borax, gave a refraction figure of 55.5 at 12.5° C., corresponding to 92.0 % of anhydrous glycerin in the liquid part, and 86.4 % in the glycerin of borax. To this figure the 11.6 % of borax present was added, making 98.0 %,

the remaining 2.0 % being water not due to the borax, but, in this case, to the glycerin.

The glycerin was also determined by *Hehner's bichromate method* (*S.P.A.*, 1887, **12**, 44 ; cp. *Analyst*, 1911, **36**, 319), convenient quantities being about 0.25 gm. with 30 ml. bichromate solution (1 ml. = 0.01 gm. glycerin); the amount found by this method was 85.8 %, an agreement sufficiently close to the previous one for practical purposes.

PROSECUTIONS. *Willesden.* Borax 5.35 %. The explanation offered was incomplete solution of the borax. Fine 10s. (*P.J.*, 1912, April 27).

Birmingham. Borax (B.P.) 18 %, glycerin (B.P.) 64 %, water 18 %. Fine £2 (1914 Report).

LIQUID EXTRACT OF CASCARA SAGRADA

This preparation first appeared in the B.P. of 1885 ; the method of preparation was unsatisfactory and was amended in 1898 and 1914. In each case the drug was extracted with water and alcohol added as a preservative.

Correspondence in the *Chemist and Druggist* in December 1900, and the next month, indicated that the drug itself yielded about 25-29 % of water-soluble matter, and that the liquid extract averaged about 22-25 w/v of solid matter, though a selected well-matured bark might yield 28 w/v. A preparation preserved with glycerin instead of alcohol had 53.8 w/v. Although the 1914 B.P. directs the presence of 25 w/v of 90 v/v alcohol, filtration is permitted which may reduce both the amount of alcohol and solid extract.

PROSECUTION. *Edinburgh.* Destitute of alcohol, of which at least 17 % should have been present. According to the evidence, the article sold contained the full proportion of the bark, but had been preserved with glycerin instead of alcohol. It was a well-known preparation which had been in use three years before the B.P. article, and was more expensive. The sheriff dismissed the case, holding that the purchaser was not prejudiced, as he knew the article was sold as "Duncan's extract." It was labelled as such, and there had been a statement made when a previous informal sample had been bought. He also observed that it was the first attempt to enforce the use of a preservative ordered by the B.P., and was afraid such prosecutions might hinder pharmaceutical research, and that special labelling might be a hindrance. The sale of the article would have been perfectly legitimate before 1885 (*C. & D.*, 1906, May 12, June 2, 16 ; *B.F.J.*, 1906, 173).

COMPOUND DECOCTION OF ALOES

According to *Cripps* (*B.P. Conf.*, 1905, 447), the 1898 preparation should have contained about 6 w/v of extractive matter and about

30 % of proof spirit. He found that some wholesale drug houses had supplied a concentrated preparation 1 part of which mixed with 3 parts of water was stated to represent the B.P. decoction. If a concentrated decoction was used, it was necessary to dilute with spirit and water, not water alone. The 1914 B.P. gave an altered formula.

H. W. Jones (*P.J.*, 1906, March 3) pointed out that the alkalinity of the ash is partly due to other constituents than the potassium carbonate.

PROSECUTIONS. *Bournemouth.* Alcohol 76 % deficient, extract of aloes 70 % deficient and potassium carbonate 50 % deficient. The defendant admitted having made a mistake and was ordered to pay costs only (*P.J.*, 1904, July 30).

Bournemouth. Alcohol 59 % deficient, extract of aloes 20 % deficient. The article was stated to have been made from the concentrated decoction. The vendor was ordered to pay costs (*P.J.*, 1904, Aug. 6).

London, South-Western. Alcohol, extract of Barbados aloes and liquorice, 55 % deficient. Fine £1 (*P.J.*, 1908, June 20).

HYPODERMIC INJECTION OF MORPHIA

PROSECUTIONS. *Kensington.* Deficient in strength 42 %. The article was dispensed from a prescription. Fine £2 (*C. & D.*, 1897, June 26).

Kensington. Acetate of morphia 25 % deficient. Although the sample was made while the inspector waited, the defence was that the article did not keep. Fine 5s. (*C. & D.*, 1898, Feb. 19).

SOLUTION OF AMMONIUM ACETATE

The 1914 B.P. directs that a litre of this solution shall contain 162.5 ml. of acetic acid, and 50 gm. or a sufficient quantity of ammonium carbonate. Sage (*P.J.*, 1927, Jan. 8) has calculated that 10 ml. distilled with NaHO should yield enough ammonia to neutralise at least 9.3 ml. N acid, but points out that the directions of the B.P. to neutralise are not sufficiently definite to settle a standard.

PROSECUTION. *Clerkenwell.* Deficient in ammonium acetate to the extent of 98 %. The deficiency was ascribed to deterioration on keeping. Fine £1 and costs (*B.F.J.*, 1910, 80).

SOLUTION OF IODINE

The "Liquor Iodi" of the 1885 B.P. was a 5 w/v solution, in water, of iodine, with 7.5 w/v potassium iodide. It has not been given in the 1898 nor 1914 Pharmacopœias, but is in the British Pharmaceutical Codex as diluted solution of iodine. The only

Birmingham sample bought under this name was in 1929. It had sp. gr. 0.810, 0.8 w/v of iodine, and no solid residue, being free from potassium iodide. The solvent used was isopropyl alcohol. It was said to have contained 1.25 w/v of iodine when prepared. The vendor was cautioned, as the article was very different from the above standard, and barely coloured the skin when used as a paint.

PROSECUTION FOR SOLUTION OF IODINE. *North London.* Iodine deficient 45.2 %. The defendant suggested that the sample was divided before solution was complete. Fine 1s. and 5 guineas costs (*F. & S.*, 1895, April 27).

CHAPTER XXXVIII

DISPENSING

Possible errors. Selection of prescription. Mixtures, bottles, insoluble powders, potassium iodide and sarsaparilla, senega. Powders, grey powder, acetylsalicylic acid. Accuracy in dispensing. Prosecutions.

DURING 1905-13, of the "prescriptions" examined in England and Wales, 15·6 % were condemned, and 17·6 % of those during 1920-30.

During 1897-1930, 358 samples of dispensed medicines were examined in Birmingham, and 112 of them were condemned; all but one had at least 10 % error. It should be explained that there are duplicates in the adulterated samples, as in a number of cases proceedings were not taken till a *second* adulterated sample had been received. Qualified pharmacists were not responsible for all the samples; one prescription dispensed at an oil, drug, and colour stores contained only 155 grains of potassium iodide, instead of the 240 grains ordered, and no label was put on the bottle! As the result of fourteen prosecutions, fines amounting to £57 were inflicted.

On the general question of testing the accuracy of dispensing, papers by Anderson and Elsdon (*B.P. Conf.*, 1920, 478) and Self and Corfield (*P.J.*, 1928, Feb. 18) should be consulted.

When a patient receives a bottle of medicine from a pharmacist he does not realise the number of possible errors from which the skill and reliability of the dispenser guards him. The following are examples:—

(1) The prescription may be misread owing to carelessness or the obscurity of the writing. (2) The prescription may be correctly read, but a lapse of memory may produce an error in the nature or quantity of the ingredients, or a stock solution may be incorrectly dispensed. (3) A neighbouring bottle with a similar label may be used instead of the correct one. (4) The weights taken may be incorrect, or a wrong quantity may be measured; one dispenser weighed paper with his iodide of potassium. (5) The correct quantities of the ingredients may be put in a bottle of the wrong size. In one case a 12-oz. bottle was used instead of a 10-oz. one; in another a 7-oz. bottle was filled instead of an 8-oz. one. (6) The label may be incorrect—*tablespoonful* being put instead of *teaspoonful*. The above mistakes will all be unintentional; but occasionally, and happily rarely, (7) a cheaper drug has been used than the one ordered, or (8) only a proportion of the expensive ingredient has

been included. There are also possible errors for which the dispenser may not be directly responsible. (9) A stock solution of a soluble drug may have been incorrectly made, avoirdupois ounces being used instead of apothecaries'. The solution may have deteriorated during keeping, or been used before the solids put in it had dissolved. (10) The drug obtained from a wholesale dealer may have been of incorrect strength (solution of ferric chloride), or have altered on keeping (quinine sulphate).

The graduation marks on a bottle may be incorrect, or a bottle graduated in *tablespoonfuls* may be given when the dose is a *teaspoonful*.

Any of the above mistakes may prevent the patient having exactly the medicine intended by his doctor, but sometimes serious results may follow a mistake. On one occasion liquid extract of *nux vomica* was dispensed instead of liquid extract of *ergot* from a neighbouring bottle. In another case 32 grains of iodide of potassium would have been taken by following the directions, instead of the $7\frac{1}{2}$ grains ordered; 3 grains of quinine sulphate in a bottle instead of the 32 grains ordered would not have had the required effect.

The debatable question as to the meaning of the sign \mathfrak{Z} has been left open by the B.P. as meaning either 480 or 437.5 grains, with the recommendation that the sign should not be used.

SELECTION OF PRESCRIPTION. The ideal prescription should be commonplace, and should not be remarkable in the ingredients, the dose, or the amount of medicine ordered, though there must be enough for analysis. Large doses will expose the purchaser to awkward questions being asked by careful pharmacists, or even a refusal to dispense the prescription, as happened when maximum doses of quinine sulphate were ordered. In a National Health Insurance enquiry the most frequently prescribed drugs were found to be sodium bicarbonate, sodium salicylate, and glycerin, each of which being prescribed four times as frequently as any other drug (Tocher, *P.J.*, 1923, Feb. 17).

The analyst should be consulted as to the suitability for analysis of a proposed prescription. It is better that the prescription be written by a doctor. National Health Insurance scripts are often used as tests, but ordinary prescriptions should also be dispensed for the protection of persons who do not come under that Act.

MIXTURES. It is a convenient, and not uncommon practice, to put the ingredients of a medicine in a bottle and fill up with the water, or other diluent, assuming that the bottle is of the correct size. In many cases there will be little error, but bottles may be seriously incorrect. Of thirty-five consecutive 8-oz. mixtures, three were $8\frac{1}{4}$ oz., twenty-two were $8\frac{1}{4}$ to $7\frac{3}{4}$ oz., seven were $7\frac{1}{2}$ oz., two were $7\frac{1}{4}$ oz., and one 7 oz. Stannard (*P.J.*, 1925, Oct. 17) examined six bottles each of seven different sizes, with the following results:—

ERROR IN DISPENSING BOTTLES (Stannard)

Error, %	0-	3-	6-9	9.5	15	Total.
No. of bottles, 1, 2, and 3 oz.	7	6	3	1	1	18
„ 4, 6, 8 and 12 oz.	15	5	4	0	0	24

The graduations of three each 8-oz. and 16-oz. bottles were examined, and seven of the forty-five, which were not top ones, were over 10 % out.

In order that the amount of the drugs actually present should be known on analysis, the Council of the Society of Public Analysts recommended (*S.P.A.*, 1923, 48, 492)—and the Ministry of Health accepted the recommendation in a circular—that inspectors should be instructed to mark the height of the contents on the bottle supplied by a vendor. The analysis can then be expressed on the actual volume, instead of that ordered.

INSOLUBLE POWDERS. It has sometimes been stated that prescriptions ordering insoluble powders are unsuitable as tests, as they cannot be correctly divided. Experience has shown that if such mixtures are thoroughly shaken several times during division, there is little difference in the three samples. With heavy magnesium carbonate the figures were 38, 38, and 44 grains per bottle, with the light carbonate, 45, 47, and 49 grains. The differences include those due to analysis as well as to sampling. With 8-oz. mixtures containing bismuth carbonate, samples from four different shops had 389–396 grains when 400 grains were ordered, and from another four shops 150–169 grains when 160 grains were ordered. Some allowance must be made in such cases, but, with careful sampling, there is no difficulty in deciding if there has been reasonably careful dispensing.

POTASSIUM IODIDE AND SARSAPARILLA MIXTURE. The amount of halogen, expressed as potassium iodide, in this mixture as dispensed by six Birmingham pharmacists varied 2.66–3.09 w/v, the theory being 2.57 w/v. These figures suggested that sarsaparilla contained chloride, and such was found to be the case (see Liverseege, *B.P. Conf.*, 1924, 752).

SENEGA MIXTURE. Prescriptions ordering 40 grains of ammonium carbonate in 8 oz. of infusion of senega were taken to twelve Birmingham pharmacists to be dispensed. The ammonium carbonate varied from not more than 34 grains to at least 70 grains per bottle, according to the minimum or maximum standard being used. Glycerin was present in two samples; the other ten had 0.40–1.36 w/v of total solids; the standard taken was 1.2 w/v. The low solids were found to be due to weak concentrated infusion having been used, and two vendors were fined. When received, titration using methyl orange, before and after distillation, indicated practically the same amount of ammonium carbonate. After keeping six weeks, two samples, owing to the development of

acidity, lost about 10 % of their alkalinity, but only about 2 % of ammonia when tested by distillation (see Liverseege, *B.P. Conf.*, 1928 ; *Q.J.P.*, 1928, 378 ; *Analyst*, 1928, 53, 499).

KEEPING PROPERTIES. Bodsworth (*P.J.*, 1929, Feb. 2) has experimented on the keeping properties of five different classes of dispensed medicines.

POWDERS. For correct dispensing each powder should be separately weighed, but sometimes the total amount has been weighed and then subdivided without separate weighing. The following are two examples showing the variation in weight :—

GREY POWDER WITH MAGNESIUM CARBONATE, 5 grains

Weight in grains	. 2.6-3.8	4.2-	4.5-	4.9-	5.2-5.3	5.6-6.6	Total.
No. of powders	. 11	6	18	24	7	5	71

The eleven lightest powders were all supplied by one pharmacist, the average weight being only 3.4 grains instead of 5 grains. The average weight of another vendor's powders was 5.1 grains, but they ranged in weight from 4.26 grains, probably through careless subdivision of the total weight.

ACETYSALICYLIC ACID, 10 grains

Weight in grains	. 8.9-9.3	9.4-	9.8-	10.2-	10.6-	11-11.7	12.0, 12.5	Total.
Powders from three vendors	. 0	10	13	1	0	0	0	24
„ „ „	. 3	8	2	2	3	4	2	24

The twenty-four powders of the first three vendors only varied from 9.4 to 10.3 grains, but those from the second three from 8.9 to 12.5 grains !

ACCURACY IN DISPENSING. Weights and measures have often been blamed as the cause of incorrect dispensing, and some experiments were made to test this. Reasonable care was aimed at, rather than strict accuracy.

2 gm. (about 31 grains) of sulphate of iron crystals were weighed on hand dispensing scales, and the accuracy of the weighing was checked by the analytical balance. The errors of three weighings were $\frac{1}{20}$, $\frac{1}{4}$ and $\frac{1}{2}$ grain. A similar experiment in which 1 to 8 gm. (15 to 124 grains) of granular sulphate of iron were weighed gave errors varying from $\frac{1}{60}$ to $\frac{1}{3}$ grain in eight weighings, the largest error being obtained when 62 grains were weighed. Granular sulphate of iron is more finely divided than the crystals used at first, and for that reason the error is less. When 1 gm. of quinine sulphate, which is a light powder, was weighed, errors of $\frac{1}{6}$ to $\frac{1}{12}$ grain were made.

160 minims of dilute acid were measured in a $\frac{1}{2}$ -oz. conical glass measure and two trials gave 160 and 162 minims. In the same measure 120 minims were measured and then 40 ; I twice obtained 159 minims, showing that no serious error was introduced by

obtaining the quantity at two measurements. Quantities of 1, 2, and 3 drachms and 1 oz. were measured in a 2-oz. conical glass measure. In six of the eight measurements the error was less than 2 %. One measurement of 1 drachm gave an error of 4 %, and one measurement of 3 drachms gave an error of 3 %. For these quantities this measure is less suitable than the smaller one.

There has been discussion as to the amount of error allowable in dispensing, and probably 10 % above or below is a fair allowance in many cases (cp. p. 45). There should be greater accuracy in weighing than in measuring. It is an advantage to have several copies of a prescription sent out simultaneously; comparison of the resulting medicines will often show what is a reasonable allowance for error. Reference has been previously made (p. 45) to the necessity of considering the range of composition given by the B.P., and that an incorrect medicine may be due to the drugs used rather than to the carelessness of the dispenser (p. 547).

See also an address on the subject by J. Butler (*P.J.* and *C. & D.*, 1932, Feb. 20).

PROSECUTIONS IN BIRMINGHAM. Quinine sulphate 23 grains in 6 oz., instead of 240 grains. The vendor said that he had reduced the quantity, as that ordered was dangerous. Fine £2 (1904 Report).

Potassium iodide 334 grains in 5½ oz., instead 360 grains in 6 oz. The dose ordered was one *teaspoonful* (= 7½ grains of potassium iodide), but the bottle was labelled "one *tablespoonful*" (= 32 grains). It had been dispensed by an unqualified assistant. Vendor paid costs (1904 Report).

Quinine sulphate 168 grains, and diluted sulphuric acid 210 minims, in 6 ozs., instead of 240 grains and 240 minims, respectively. The vendor attributed the deficiency to the quinine sulphate being weighed on paper without a corresponding counterpoise on the other scale pan. Paid costs. The medicine as dispensed at another shop had only 127 grains of quinine sulphate and 210 minims of diluted sulphuric acid. The vendor interfered with the division of the sample and was fined £2 for obstruction (1913 Report).

Quinine sulphate 19 grains and diluted hydrobromic acid 206 minims in 8 oz., instead of 32 grains and 240 minims, respectively. The deficiency was attributed to the stock mixture being diluted to 100 oz. instead of 80 oz.; the label marking the volume of 80 oz. having come off. Possibly some quinine sulphate had been blown away after weighing. The use of stock solutions was not allowed by the N.H.I. Committee. Fine £15 (1921 Report).

"White mixture" containing only 72 % of the proper amount of magnesium sulphate, and 62 grains of *light* magnesium carbonate in 8 oz., instead of 240 grains of *heavy* magnesium carbonate. Fine £5.

The vendor had been cautioned three months before for incorrect dispensing, and between the two samples being taken had declined to dispense another prescription, saying he was "out of stock" of common chemicals. He had recognised the inspector (1922 Report).

Ammonium carbonate at least 50 grains in 7.9 oz. of weak infusion of senega, with 14 grains of solid extract. The prescription ordered 40 grains of ammonium carbonate in 8 oz. of infusion of senega, and at least 42 grains of solid extract should have been present. Fine £1. An informal sample dispensed five days previously had a similar composition (1928 Report).

PROSECUTION AT ISLINGTON. Quinine sulphate 25 % in excess of the quantity ordered in the mixture. The defendants attributed the excess found to the quinine sulphate having lost almost all its water of crystallisation. Fine £5 (*Analyst*, 1925, **50**, 184).

APPENDIX

STANDARDISATION OF VOLUMETRIC APPARATUS

For many analytical purposes it is of little importance if the millilitre, the cubic centimetre, or Mohr's unit (G.W.A.), which is the volume of 1 gm. of water at 17.5° C. (the relation being 1,000 : 1,000.027 : 1002), be used as the unit, if all the measurements and standard solutions are *made on the same basis*. In each of the cases the *relation* is correct. If, however, *absolute* measurements are required, such as the w/v of solid matter in a B.P. liquid preparation, the millilitre basis only should be used (cp. *Analyst*, 1924, **49**, 479).

The National Physical Laboratory, when testing pipettes, prescribes that pipettes shall be allowed to drain fifteen seconds in contact with the side of the containing vessel after the outflow has ceased. In ordinary practice shortening the time of draining to five or ten seconds will introduce little error, but the selected time must be adhered to.

Pipettes, flasks, and burettes should be tested on receipt to ascertain if the errors are within those allowed by the National Physical Laboratory (see *Analyst*, 1924, **49**, 477). For pipettes the water contents at a definite temperature should be weighed, and a correction made for temperature. Standard pipettes may be used for testing flasks containing not more than 250 ml. Examples of errors which have been found in pipettes may be found in a paper by the writer (*B.P. Conf.*, 1894, 481). Foulk (*Analyst*, 1915, **40**, 455), has described an apparatus for testing burettes, Coste (*S.P.A.*, 1917, **42**, 385) has given a note on burette jets, and More (*S.P.A.*, 1929, **54**, 630) has discussed meniscus corrections.

For the Board of Trade allowances for error in apothecaries' graduated measures, see *P.J.*, 1929, June 29.

PREPARATION OF "E" TEST SOLUTIONS

For a number of analytical methods it is advisable to use acid, alkaline, or other solutions in definite quantities, or of definite strength, but the accuracy of strength required is not sufficient to justify using standard volumetric solutions. Further, there is no simple relationship in chemical action between percentage solutions such as are often used for testing. For example, 10 % solutions of different acids are not of the same acidity, and will not neutralise 10 % solutions of alkalis. For simplicity and convenience Reddrop recommended (*Chem. News*, 1890, May 23, 30) preparing test solutions on an approximately normal basis, and the use of the symbol "E" (Equivalent) to distinguish such solutions from accurate normal solutions. Two other advantages may be mentioned. Some B.P. acids may vary in strength between limits, and therefore cannot be expressed in terms of Normal, though their "E" values can be usefully given. Some reagents are described in terms of sp. gr., a statement which is less convenient than "E" value. Some salts, as phosphates, differ in normal value according to the indicator used; in such cases the use of "M" for molecular weight in grammes per litre is better than "E." The calculation of the dilution of "E" solutions has been given previously (p. 108).

TABLE I

ATOMIC WEIGHTS (1932), ALSO COMPOUNDS AND MULTIPLES

Substance	Weight	$\times 2$	$\times 3$	Other Multiples
Aluminium .	26.97	53.94		
Antimony .	121.76	243.52		
Arsenic .	74.93	149.86		
Barium .	137.36	274.72		
BaSO ₄ .	233.42	466.84	700.26	4 = 933.68
Bismuth .	209.00	418.00		
Boron .	10.82	21.64		
Bromine .	79.916	159.832		
Calcium .	40.08	80.16	120.24	
Carbon .	12.00			
C ₂ H ₅ O ₂ .	59.0234	118.0468		
C ₇ H ₅ O ₂ .	121.039			
CO ₂ .	60.00	120.00		
C ₆ H ₅ O ₇ .	189.039			
C ₃ H ₅ O ₃ .	89.039	178.078		
C ₂ O ₄ .	88.00			
C ₇ H ₅ O ₃ .	137.039			
C ₄ H ₄ O ₆ .	148.031			
Chlorine .	35.457	70.914	106.371	
Chromium .	52.01	104.02		
CrO ₄ .	116.01			
Cr ₂ O ₇ .	216.02			
Copper .	63.57	127.14		
Hydrogen .	1.0078	2.0156	3.0234	4 = 4.0312, 5 = 5.039, 6 = 6.0468
H ₂ O .	18.0156	36.0312	54.0468	4 = 72.0624, 5 = 90.078, 6 = 108.0936
Iodine .	126.932	253.864		
Iron .	55.84	111.68	167.52	
Lead .	207.22	414.44	621.66	
Lithium .	6.940	13.880		
Magnesium .	24.32	48.64	72.96	4 = 97.28
Manganese .	54.93	109.86	164.79	
Mercury .	200.61	401.22		
Nitrogen .	14.008	28.016	42.024	
NH ₄ .	18.0392	36.0784	54.1176	
Oxygen .	16.0	32.0	48.0	
Phosphorus .	31.02	62.04		
PO ₄ .	95.02	190.04	285.06	4 = 380.08
Platinum .	195.23	390.46		
Potassium .	39.10	78.20	117.30	
Silicon .	28.06	56.12		
Silver .	107.880	215.760		
AgCl .	143.337	286.674	430.011	
Sodium .	22.997	45.994	68.991	
Strontium .	87.63			
Sulphur .	32.06	64.12	96.18	
SO ₄ .	96.06	192.12	288.18	4 = 384.24
Tin .	118.70	237.40		
Uranium .	238.14	476.28		
Zinc .	65.38	130.76		

TABLE II
CONVERSION FACTORS
(Arranged in order of bases)

To convert A to B, multiply by	A	B	To convert B to A, multiply by
1.890 . . .	2Al	Al ₂ O ₃	0.5291
0.1124 . . .	Al ₂ (NH ₄) ₂ (SO ₄) ₄ , 24 H ₂ O	Al ₂ O ₃	8.894
1.034 . . .	„	4BaSO ₄	0.9710
0.03757 . . .	„	2NH ₃	26.617
0.1074 . . .	Al ₂ K ₂ (SO ₄) ₄ , 24 H ₂ O	Al ₂ O ₃	9.307
0.9841 . . .	„	4BaSO ₄	1.0161
12.308 . . .	2NH ₄	(NH ₄) ₂ PtCl ₆	0.08125
0.8775 . . .	NH ₄ C ₇ H ₅ O ₂	HC ₇ H ₅ O ₂	1.140
0.5601 . . .	N ₃ H ₁₁ C ₂ O ₅	2CO ₂	1.7854
1.569 . . .	As ₂ O ₃	Mg ₂ As ₂ O ₇	0.6373
1.115 . . .	2Bi	Bi ₂ O ₃	0.8970
0.8979 . . .	(Bi ₂ O ₂ CO ₃) ₂ , H ₂ O	2Bi ₂ O ₃	1.114
0.7639 . . .	2(BiONO ₃ , H ₂ O)	Bi ₂ O ₃	1.309
0.6436 . . .	2(BiOC ₇ H ₅ O ₃)	„	1.554
0.5630 . . .	2H ₃ BO ₃	B ₂ O ₃	1.776
1.399 . . .	Ca	CaO	0.7147
0.5603 . . .	CaCO ₃	„	1.7846
0.4397 . . .	„	CO ₂	2.2745
0.6240 . . .	Ca(C ₃ H ₅ O ₃) ₂	CaSO ₄	1.6025
0.4417 . . .	Ca(C ₃ H ₅ O ₃) ₂ , 5H ₂ O	„	2.264
0.7568 . . .	Ca(OH) ₂	CaO	1.321
0.3257 . . .	CaSO ₄ , 2H ₂ O	„	3.070
0.7907 . . .	„	CaSO ₄	1.2646
1.3558 . . .	„	BaSO ₄	0.7376
4.043 . . .	Cl	AgCl	0.2474
3.931 . . .	HCl	„	0.2544
1.252 . . .	Cu	CuO	0.7989
0.3186 . . .	CuSO ₄ , 5H ₂ O	„	3.139
1.112 . . .	Cu ₂ O	„	0.8994
0.3998 . . .	C ₂ H ₅ NO ₂	NO	2.501
1.850 . . .	I	AgI	0.5406
1.835 . . .	HI	„	0.5449
1.088 . . .	ICl ₃	2I	0.9190
1.430 . . .	Fe ₂	Fe ₂ O ₃	0.6994
0.6892 . . .	2FeCO ₃	„	1.451
0.4820 . . .	FeCO ₃	Fe	2.074
0.4922 . . .	2FeCl ₃	Fe ₂ O ₃	2.032
0.2872 . . .	2FeSO ₄ , 7H ₂ O	„	3.482
0.5292 . . .	2FePO ₄	„	1.8896
1.077 . . .	Pb	PbO	0.9283
1.831 . . .	„	Pb(C ₂ H ₃ O ₂) ₂ , 3H ₂ O	0.5463
1.251 . . .	PbSO ₄	„	0.7996
1.5985 . . .	„	Pb(NO ₃) ₂	0.6256
1.464 . . .	„	PbSO ₄	0.6833
4.578 . . .	Mg ₂	Mg ₂ P ₂ O ₇	0.2184
2.7614 . . .	2MgO	„	0.3621
1.219 . . .	(MgCO ₃) ₃ , Mg(OH) ₂ , 3H ₂ O	2Mg ₂ P ₂ O ₇	0.8204
0.4414 . . .	„	4MgO	2.265
0.3613 . . .	„	3CO ₂	2.768
1.162 . . .	(MgCO ₃) ₃ , Mg(OH) ₂ , 4H ₂ O	2Mg ₂ P ₂ O ₇	0.8608
0.4207 . . .	„	4MgO	2.377
0.3443 . . .	„	3CO ₂	2.904
0.4517 . . .	2(MgSO ₄ , 7H ₂ O)	Mg ₂ P ₂ O ₇	2.214

CONVERSION FACTORS

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To convert A to B, multiply by		To convert B to A, multiply by	
	A		B
0.9470	$\text{MgSO}_4, 7\text{H}_2\text{O}$	1.056	BaSO_4
1.160	Hg	0.8622	HgS
1.074	HgO	0.9310	"
0.8569	HgCl_2	1.167	"
0.9857	HgCl	1.015	"
0.9230	NH_2HgCl	1.083	"
0.5120	HgI_2	1.953	"
1.216	N	0.8225	NH_3
1.288	"	0.7765	NH_4
2.142	"	0.4668	NO
2.058	NH_3	0.4860	NH_4OH
2.290	2P	0.4368	P_2O_5
1.032	PO_4	0.9692	H_3PO_4
0.7474	2PO_4	1.338	P_2O_5
3.590	2P	0.2786	$\text{Mg}_2\text{P}_2\text{O}_7$
1.172	2PO_4	0.8534	"
1.568	P_2O_5	0.6379	"
1.380	"	0.7243	$2\text{H}_3\text{PO}_4$
2.184	"	0.4578	$\text{Ca}_3(\text{PO}_4)_2$
1.649	"	0.6066	$\text{CaH}_4(\text{PO}_4)_2$
2.268	"	0.4410	$\text{CaNa}_4(\text{PO}_4)_2$
1.831	"	0.5461	$\text{CaNa}_2\text{P}_2\text{O}_7$
1.958	"	0.5107	$\text{CaHPO}_4, \text{Na}_2\text{HPO}_4$
1.395	"	0.7170	CaP_2O_6
6.217	2K	0.1609	K_2PtCl_6
2.228	"	0.4488	K_2SO_4
1.2046	"	0.8302	K_2O
0.5245	KCl	1.907	K
0.4752	"	2.104	Cl
0.7841	"	1.275	NaCl
3.260	2KCl	0.3068	K_2PtCl_6
1.414	KI	0.7071	AgI
0.1385	KNO_3	7.217	N
0.2976	"	3.370	NO
0.7704	$\text{K}_2\text{C}_2\text{H}_4\text{O}_6$	1.298	K_2SO_4
0.4631	$2\text{KHC}_4\text{H}_4\text{O}_6$	2.159	"
1.3395	K_2SO_4	0.7465	BaSO_4
3.088	2Na	0.3238	Na_2SO_4
1.348	"	0.7419	Na_2O
0.8473	$\text{NaC}_7\text{H}_5\text{O}_2$	1.180	$\text{HC}_7\text{H}_5\text{O}_2$
0.8455	2NaHCO_3	1.183	Na_2SO_4
0.5238	NaHCO_3	1.909	CO_2
0.6309	2NaHCO_3	1.585	Na_2CO_3
0.4151	Na_2CO_3	2.409	CO_2
0.3704	$\text{Na}_2\text{CO}_3, 10\text{H}_2\text{O}$	2.700	Na_2CO_3
0.6486	$\text{Na}_2\text{B}_4\text{O}_7, 10\text{H}_2\text{O}$	1.542	$4\text{H}_3\text{BO}_3$
0.5277	"	1.895	$\text{Na}_2\text{B}_4\text{O}_7$
0.6068	NaCl	1.648	Cl
1.215	2NaCl	0.8230	Na_2SO_4
1.566	NaI	0.6385	AgI
0.8626	$\text{NaC}_7\text{H}_5\text{O}_3$	1.159	$\text{HC}_7\text{H}_5\text{O}_3$
1.643	Na_2SO_4	0.6086	BaSO_4
1.533	SiO_2	0.6524	SiO_4
7.281	S	0.1374	BaSO_4
3.644	SO_2	0.2745	"
2.9155	SO_3	0.3430	"
2.430	SO_4	0.4115	"
2.380	H_2SO_4	0.4202	"
1.270	Sn	0.7877	SnO_2

To convert A to B, multiply by	A	B	To convert B to A, multiply by
1.245 . . .	Zn	ZnO	0.8034
0.7135 . . .	$\text{ZnCO}_3(\text{Zn}2\text{OH})_2, \text{H}_2\text{O}$	3ZnO	1.402
0.5971 . . .	ZnCl_2	ZnO	1.675
0.2830 . . .	$\text{ZnSO}_4, 7\text{H}_2\text{O}$	„	3.533

TABLE III

VOLUMETRIC FACTORS

(Arranged in order of acids and alkalies)

It is suggested that titrations should be calculated to N. v/w, or N. v/v, and the appropriate factor then used.

Substance	Formula	Molecular Weight	1 ml. Normal = gm.	
Acetate . . .	$\text{HC}_2\text{H}_3\text{O}_2$	60.031	.0600	
	$\text{C}_2\text{H}_3\text{C}_2\text{H}_3\text{O}_2$	88.06	.0881	
	$\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2, 3\text{H}_2\text{O}$	379.32	.1897	N/1036, 1 ml. = .000183 After ignition.
	$\text{KC}_2\text{H}_3\text{O}_2$	98.123	.09812	
	$\text{UO}_2(\text{C}_2\text{H}_3\text{O}_2)_2, 2\text{H}_2\text{O}$	424.22	.1413	
Acetylsalicylic acid . . .	$\text{C}_9\text{H}_8\text{O}_4$	180.06	.1801	
Ammonia . . .	NH_3	17.031	.01703	
	NH_4OH	35.047	.03505	
	Sb_2O_3	291.52	.0729	
Antimony . . .	As_2O_3	197.87	.04947	
Arsenate . . .	Na_2HAsO_4	185.93	.09297	
	$\text{Ba}(\text{OH})_2$	171.38	.0857	
Baryta . . .	$\text{HC}_7\text{H}_5\text{O}_2$	122.047	.1220	
Benzoate . . .	$\text{NH}_4\text{C}_7\text{H}_5\text{O}_2$	139.07	.1391	
	$\text{NaC}_7\text{H}_5\text{O}_2$	144.036	.1440	
	$\text{K}_2\text{Cr}_2\text{O}_7$	294.22	.04904	
Bichromate . . .	H_3BO_3	61.843	.06184	
Borate . . .	B_2O_3	69.64	.03482	
	$\text{Na}_2\text{B}_4\text{O}_7, 10\text{H}_2\text{O}$	381.43	.1907	
	Br	79.916	.0799	N/70.91, 1 ml. = .001127 „ „ .001141 „ „ .001381 „ „ .001678 „ „ .001451 „ „ .002507
Bromide . . .	HBr	80.924	.0809	
	NH_4Br	97.955	.09795	
	KBr	119.02	.1190	
	NaBr	102.913	.1029	
	$\text{SrBr}_2, 6\text{H}_2\text{O}$	355.56	.1778	
Butyric acid . . .	$\text{HC}_4\text{H}_7\text{O}_2$	88.062	.0881	
Calcium . . .	CaO	56.08	.02804	
	$\text{Ca}(\text{OH})_2$	74.0956	.03705	
Carbonate . . .	Co_2	44.00	.022	
	$(\text{NH}_4)_2\text{CO}_3$	96.078	.04804	
	NH_4HCO_3	79.047	.07905	
	$\text{N}_3\text{H}_{11}\text{C}_2\text{O}_5$	157.11	.05237	
	CaCO_3	100.0836	.05005	
	$\text{Ca}(\text{HCO}_3)_2$	162.09	.08106	
	FeCO_3	115.84	.1158	
	Li_2CO_3	73.88	.03694	
	$(\text{MgCO}_3)_3, \text{Mg}(\text{OH})_2, 3\text{H}_2\text{O}$	365.34	.04587	
	$(\text{MgCO}_3)_3, \text{Mg}(\text{OH})_2, 4\text{H}_2\text{O}$	383.36	.04791	
	K_2CO_3	138.20	.0691	
	KHCO_3	100.108	.1001	
	Na_2CO_3	105.994	.0530	
	$\text{Na}_2\text{CO}_3, 10\text{H}_2\text{O}$	286.15	.1432	
	NaHCO_3	84.005	.0840	
	$\text{Mg}(\text{HCO}_3)_2$	146.34	.07317	
Cerotic acid . . .	$\text{HC}_{16}\text{H}_{31}\text{O}_2$	396.40	.3964	

Substance	Formula	Molecular Weight	1 ml. Normal = gm.	
Chloride .	Cl	35.457	·03546	N/70.91, 1 ml. = ·0005
	HCl	36.465	·03646	" " ·000514
	NH ₄ Cl	53.496	·0535	" " ·000754
	C ₂ H ₅ Cl	64.496	·0645	
	FeCl ₂	126.75	·1268	
	NH ₂ HgCl	252.09	·126	
	KCl	74.557	·07456	N/70.91, 1 ml. = ·00105
	NaCl	58.454	·05845	" " ·00824
	C ₂₀ H ₂₄ N ₂ O ₂ (HCl) ₂	397.113	·1986	
Citrato .	H ₃ C ₆ H ₅ O ₇ , H ₂ O	210.08	·0700	
	Li ₃ C ₆ H ₅ O ₇ ·4H ₂ O	281.92	·09397	After ignition.
	K ₃ C ₆ H ₅ O ₇ , H ₂ O	324.35	·1081	" "
	Na ₃ C ₆ H ₅ O ₇	258.03	·0860	" "
Cyanide	HCN	27.015	·054	
	KCN	65.11	·1302	
Formaldehyde	CH ₂ O	30.016	·0300	
Hexamino	C ₆ H ₁₂ N ₄	140.12	·0350	
Hydrogen peroxide	H ₂ O ₂	34.016	·0170	
Iodine .	I	126.932	·1269	N/18.13, 1 ml. = ·007
	HI	127.94	·1279	N/70.91, " ·00179
	ICl	162.389	·08119	" " ·00180
	FeI ₂	309.70	·1548	Wijs solution is about N/5
	KI	166.032	·166	N/70.91, 1 ml. = ·002341
	NaI	149.929	·1499	" " ·002114
Iron .	Fe	55.84	·05584	
	FeO	71.84	·07184	
Lactate .	H C ₃ H ₅ O ₃	90.05	·09005	
	Ca(C ₃ H ₅ O ₃) ₂	218.16	·1091	
	Ca(C ₃ H ₅ O ₃) ₂ ·5H ₂ O	308.24	·1541	
Lead .	Pb	207.22	·1036	N/1036, 1 ml. = ·0001
Magnesia	MgO	40.32	·02015	
Permanganate	KMnO ₄	158.03	·03161	
Mercury .	HgO	216.61	·1083	
Myricin .	C ₃₆ H ₃₁ O ₂	676.75	·06768	
Nitrogen	HNO ₃	63.016	·06302	
	Pb(NO ₃) ₂	331.24	·1656	N/1036, 1 ml. = ·00016
	AgNO ₃	169.89	·1699	N/70.91, 1 ml. = ·00239
	HNO ₂	47.016	·0235	
	NaNO ₂	69.005	·0345	
Oleic acid	H C ₁₈ H ₃₃ O ₂	282.27	·02823	
Oxalic acid	H ₂ C ₂ O ₄ ·2H ₂ O	126.047	·0630	
Palmitic acid	H C ₁₆ H ₃₁ O ₂	256.25	·0256	
Tripalmitin	C ₅₄ H ₉₈ (C ₁₆ H ₃₁ O ₂) ₃	806.77	·0269	
Phosphorus *	H ₃ PO ₄	98.043	·0527	Tribasic †
			·0490	Dibasic ‡
			·0980	Monobasic §
	PO ₄	95.02	·03167	Tribasic
			·0475	Dibasic
			·0950	Monobasic
	P ₂ O ₅	142.04	·02367	Tribasic
			·0355	Dibasic
			·0710	Monobasic
	AlPO ₄	121.99	·122	"
	(NH ₄) ₂ HPO ₄	132.11	·1321	"
	CaH ₄ (PO ₄) ₂	234.14	·1171	Dibasic
	Fe ₃ (PO ₄) ₂ ·8H ₂ O	501.68	·1672	Monobasic
	NaH ₂ PO ₄	120.03	·120	"
	Na ₂ HPO ₄ ·12H ₂ O	358.21	·3582	"
	Ca(PH ₃ O ₂) ₃	170.15	·02127	
	NaPH ₃ O ₂	88.03	·0220	

* See pp. 73f.

† Phenol phthalein with CaCl₂.

‡ Phenol phthalein.

§ Methyl orange, or difference between methyl orange and phenol phthalein.

Substance	Formula	Molecular Weight	1 ml. Normal = gm.	
Picric acid	$\text{C}_6\text{H}_3(\text{NO}_2)_3\text{OH}$	229.05	.0229	
Potash	KOH	56.108	.0561	
	K_2O	94.20	.0471	
Protein *	$\text{N} \times 6.25$	87.55	.08755	
	$\text{N} \times 6.38$	89.37	.08937	
Salicylate	$\text{HC}_7\text{H}_5\text{O}_3$	138.047	.1380	
	$\text{NaC}_7\text{H}_5\text{O}_3$	160.036	.160	After ignition.
Soda	NaOH	40.005	.0400	
	Na_2O	61.994	.0310	
Stearic acid	$\text{HC}_{18}\text{H}_{35}\text{O}_2$	284.28	.284	
Tristearin	$\text{C}_{57}\text{H}_{113}\text{O}_6$	890.86	.297	
Succinic acid	$\text{H}_2\text{C}_4\text{H}_4\text{O}_4$	118.05	.059	
Sulphur	H_2SO_4	98.076	.04904	
	SO_4	96.06	.04803	
	SO_3	80.06	.0403	
	FeSO_4	151.90	.1519	
	$\text{FeSO}_4, 7\text{H}_2\text{O}$	278.01	.2780	
	$\text{FeSO}_4, (\text{NH}_4)_2\text{SO}_4, 6\text{H}_2\text{O}$	392.13	.3921	
	KHSO_4	136.168	.1362	
	H_2SO_3	82.076	.04104	
	SO_2	64.06	.03203	
	$\text{Na}_2\text{SO}_3, 7\text{H}_2\text{O}$	252.16	.1261	
	$\text{Na}_2\text{S}_2\text{O}_3, 5\text{H}_2\text{O}$	248.2	.2482	N/18.13, 1 ml. = .01369
Tartrate	$\text{H}_2\text{C}_4\text{H}_4\text{O}_6$	150.05	.07502	
	$(\text{KSbOC}_4\text{H}_4\text{O}_6)_2\text{H}_2\text{O}$	667.8	.167	
	$(\text{K}_2\text{C}_4\text{H}_4\text{O}_6)_2\text{H}_2\text{O}$	470.48	.1176	After ignition.
	$\text{KHC}_4\text{H}_4\text{O}_6$	188.14	.1881	
	$\text{KNaC}_4\text{H}_4\text{O}_6, 4\text{H}_2\text{O}$	282.19	.1411	After ignition.
	$\text{Na}_2\text{C}_4\text{H}_4\text{O}_6$	194.02	.0970	" "
	$\text{Na}_2\text{C}_4\text{H}_4\text{O}_6, 2\text{H}_2\text{O}$	230.06	.115	" "

TABLE IV

"E" TEST SOLUTIONS. (See pages 108, 552.)

The solutions marked "official" are of the strengths ordered by the Fertilisers and Feeding Stuffs Regulations, 1928.

ACIDS

$\text{HC}_2\text{H}_3\text{O}_2$	17.5 E = 105 w/v = B.P. glacial acid
	10 E = 60 w/v
	E = 6 w/v
HCl	10 E = 36.5 w/v = B.P. acid
	3 E = 11.0 w/v
	0.27 E = 1 % (official)
HNO_3	15.8 E = 99.4 w/v = B.P. acid
	5.9 E = sp. gr. 1.19 (official)
	3 E = 18.9 w/v
	0.15 E = 1 % (official)
H_2SO_4	36 E = 176 w/v = B.P. acid
	12.3 E = sp. gr. 1.35 (official)
	3.6 E = strength for Reichert
	3 E = 14.7 w/v
	0.255 E = 1.25 w/v (official)

ALKALIS

NH_3	18 E = 30 w/v = sp. gr. 0.880
	15 E = 25 w/v. This strength is safer to keep than
	18 E. Add 400 ml. water to W.Q.
	of sp. gr. 0.880

* See Broese Jones (*B.F.J.*, 1931, 116; 1932, 1, 12, etc.)

	6 E = 10 w/v	
	4.5 E = 7.7 w/v = 8 w/w (Official)	
	1.5 E = 2.5 w/v	
	1.1 E = 2 w/v (official)	
(NH ₄) ₂ CO ₃ . . .	5 E = 24 w/v.	Dissolve 19.6 gm. powdered ammonium carbonate in 21 ml. 6E.NH ₃ and 80 ml. water, and filter.
KOH . . .	10 E = 56 w/v.	Dissolve 1 lb. of 90 % sticks in 620 ml. of water, while cooling.
	0.47-0.5 E	Alcoholic potash. Dilute 5.3 ml. 10 E.KOH with purified methylated spirit to 100 ml. Filter after standing. Titrate, and dilute if necessary.
NaOH . . .	15 E = 60 w/v.	Add 1 lb. of sticks (pure by alcohol) to 600 ml. of water, while cooling. Syphon off after shaking and settling.
	10 E = 40 w/v.	Dissolve 1 lb. in 1,000 ml. of water as above.
	3 E = 12 w/v	
	0.312 E = 1.25 w/v (official)	
	2.2 E	Glycerol soda, for Reichert. Add 700 ml. of glycerol to 200 ml. 10 E.NaOH in a flask. Warm and shake to mix. 15 ml. (the measure being washed out) must be made acid by 10 ml. of 3.6 E.H ₂ SO ₄
Na ₂ CO ₃	E = 5.3 w/v	

SALTS, ETC.

C ₂ H ₅ OH . . .	16.7 M = 95 w/w (official, if "per cent." means w/w)	
	16.4 M = 95 v/v (official, if "per cent." means v/v)	
	16.1 M = 74.2 w/v = 64° O.P.	
	13.8 M = sp. gr. 0.864 (official)	
NH ₄ Cl . . .	4 E = 21.4 w/v	
(NH ₄) ₂ C ₂ O ₄ , H ₂ O .	E/2 = 3.6 w/v	
BaCl ₂ , 2H ₂ O . . .	E = 12.2 w/v	
CaCl ₂ . . .	E = 5.5 w/v of fused salt	
FeCl ₃ . . .	2.7 E = 14.7 w/v = B.P. <i>Liq. Ferri Perchlor.</i>	
	E/2 = 1.1 w/v	
Pb(C ₂ H ₃ O ₂) ₃ , 3H ₂ O	E = 19 w/v	
Pb ₂ O(C ₂ H ₃ O ₂) ₂ . . .	2.2 E = 30 w/v = B.P. <i>Liq. Plumbi Subacet. Fort.</i>	
K ₂ CrO ₄ . . .	E = 9.7 w/v	
K ₄ Fe(CN) ₆ , 3H ₂ O .	E/2 = 5.3 w/v	
K ₃ Fe(CN) ₆ . . .	E/2 = 5.5 w/v	
KI . . .	E = 16.6 w/v	
K ₂ SO ₄ . . .	E = 8.7 w/v	
KCNS . . .	E = 9.7 w/v	
Na ₂ HPO ₄ , 12H ₂ O	E = 12.0 w/v	

TABLE V

CORRECTION OF SP. GR. OF MILK FOR TEMPERATURE

(Sp. gr. × 1,000) - 1,000 . . .	20-	25-	28-	31-	33-
50-59° F. for each 1°, subtract08	.09	.10	.11	.12
61-70° F. for each 1°, add11	.12	.13	.14	.15

TABLE VI
CALCULATION OF FAT IN MILK FROM SP. GR. AND
TOTAL SOLIDS

From Richmond's formula (*S.P.A.*, 1894, **19**, 81) :—

$$F = \frac{T.S.}{1.2} - \frac{0.219 \times G}{\text{sp. gr.}}$$

F = Fat %_o. T.S. = total solids, %_o. G = (1,000 × sp. gr.) — 1,000.

G at 60° F.	Total Solids, % _o .							$\frac{0.219 \times G}{\text{sp. gr.}}$
	9.0	10.0	11.0	12.0	13.0	14.0	15.0	
20	3.20	4.03	4.86	5.70	6.53	7.36	8.20	4.30
21	3.00	3.83	4.66	5.50	6.33	7.16	8.00	4.50
22	2.79	3.62	4.45	5.29	6.12	6.95	7.79	4.71
23	2.58	3.41	4.24	5.08	5.91	6.74	7.58	4.92
24	2.37	3.20	4.03	4.87	5.70	6.53	7.37	5.13
25	2.16	2.99	3.82	4.66	5.49	6.32	7.16	5.34
26	1.95	2.78	3.61	4.45	5.28	6.11	6.95	5.55
27	1.74	2.57	3.40	4.24	5.07	5.90	6.74	5.76
28	1.53	2.36	3.19	4.03	4.86	5.69	6.53	5.97
29	1.33	2.16	2.99	3.83	4.66	5.49	6.33	6.17
30	1.12	1.95	2.78	3.62	4.45	5.28	6.12	6.38
31	0.92	1.75	2.58	3.42	4.25	5.08	5.92	6.58
32	0.71	1.54	2.37	3.21	4.04	4.87	5.71	6.79
33	0.50	1.33	2.16	3.00	3.83	4.66	5.50	7.00
34	0.30	1.13	1.96	2.80	3.63	4.46	5.30	7.20
35	0.09	0.92	1.75	2.59	3.42	4.25	5.09	7.41

Subtract 0.10 % of fat for increase of 0.5 G.
T.S. fractions, % 1 .2 .3 .4 .5 .6 .7 .8 .9
Corresponding addition to
fat, % 0.8 1.7 2.5 3.3 4.2 5.0 5.8 6.7 7.5

TABLE VII

CALCULATION OF SOLIDS-NOT-FAT IN MILK FROM SP. GR. AND FAT

From Richmond's formula (*S.P.A.*, 1894, 19, 81) :—

$$\text{S.N.F.} = \frac{0.263 \times G}{\text{sp. gr.}} + 0.2 \text{ F.}$$

F = fat %. S.N.F. = solids-not-fat %. $G = (1,000 \times \text{sp. gr.}) - 1,000$.

G at 60° F.	2.0	2.5	Fat, %. 3.0	3.5	4.0	$\frac{0.263 \times G}{\text{sp. gr.}}$
20	5.56	5.66	5.76	5.86	5.96	5.16
21	5.80	5.90	6.00	6.10	6.20	5.40
22	6.05	6.15	6.25	6.35	6.45	5.65
23	6.30	6.40	6.50	6.60	6.70	5.90
24	6.56	6.66	6.76	6.86	6.96	6.16
25	6.81	6.91	7.01	7.11	7.21	6.41
26	7.06	7.16	7.26	7.36	7.46	6.66
27	7.31	7.41	7.51	7.61	7.71	6.91
28	7.56	7.66	7.76	7.86	7.96	7.16
29	7.80	7.90	8.00	8.10	8.20	7.40
30	8.06	8.16	8.26	8.36	8.46	7.66
31	8.30	8.40	8.50	8.60	8.70	7.90
32	8.55	8.65	8.75	8.85	8.95	8.15
33	8.80	8.90	9.00	9.10	9.20	8.40
34	9.04	9.14	9.24	9.34	9.44	8.64
35	9.29	9.39	9.49	9.59	9.69	8.89

Add 0.12 % of solids-not-fat for increase of 0.5 G.

Fat fractions, % . . . 1 .2 .3 .4 .5 .6 .7 .8 .9

Corresponding additions
to solids-not-fat, % . . .02 .04 .06 .08 .10 .12 .14 .16 .18

TABLE VIII

SPECIFIC GRAVITY AND w/v ABSOLUTE ALCOHOL

Calculated from Thorpe's tables (Sp. gr. \times % by weight):—

Sp. gr.	w/v.	Sp. gr.	w/v.	Sp. gr.	w/v.	Sp. gr.	w/v.	Sp. gr.	w/v.
·820	74·5	·840	69·9	·860	64·5	·880	58·7	·900	52·3
1	·3	1	·6	1	·3	1	·4	1	51·9
2	·1	2	·4	2	·0	2	·0	2	·6
3	73·9	3	·1	3	63·7	3	57·7	3	·2
4	·7	4	68·9	4	·4	4	·4	4	50·9
·825	·5	·845	·6	·865	·1	·885	·1	·905	·6
6	·2	6	·3	6	62·8	6	56·8	6	·2
7	·0	7	·1	7	·5	7	·5	7	49·9
8	72·8	8	67·8	8	·2	8	·2	8	·5
9	·5	9	·6	9	·0	9	55·8	9	·2
·830	·3	·850	·3	·870	61·7	·890	·5	·910	48·8
1	·1	1	·0	1	·4	1	·2	1	·5
2	71·8	2	66·8	2	·1	2	54·9	2	·1
3	·6	3	·5	3	60·8	3	·6	3	47·8
4	·4	4	·2	4	·5	4	·2	4	·4
·835	·1	·855	65·9	·875	·2	·895	53·9	·915	·1
6	70·9	6	·7	6	59·9	6	·6	6	46·7
7	·6	7	·4	7	·6	7	·3	7	·3
8	·4	8	·1	8	·3	8	52·9	8	·0
9	·1	9	64·8	9	·0	9	·6	9	45·6

TABLE IX

CORRECTION OF SP. GR. OF ALCOHOL FOR TEMPERATURE

(Liverseege, *S.P.A.*, 1897, 22, 154)

Specific Gravity.	1° F. =	Specific Gravity.	1° F. =
0·794—	0·00046	0·965—	0·00026
0·864—	45	0·966—	25
0·889—	44	0·967—	24
0·902—	43	0·968—	23
0·912—	42	0·969—	22
0·921—	41	0·970—	21
0·928—	40	0·971—	20
0·935—	39	0·973—	19
0·940—	38	0·974—	18
0·943—	37	0·975—	17
0·946—	36	0·976—	16
0·949—	35	0·977—	15
0·951—	34	0·978—	14
0·953—	33	0·980—	13
0·955—	32	0·981—	12
0·957—	31	0·983—	11
0·959—	30	0·985—	10
0·961—	29	0·987—	0·00009
0·962—	28	0·990—	8
0·963—	27	0·995—1·000	7

When the temperature is lower than 60° F., the correction given, multiplied by the difference in temperature, is to be subtracted from the specific gravity found ; but when the temperature is higher, added to it.

TABLE X

QUANTITIES OF ARTICLES TO BE BOUGHT FOR ANALYSIS

ARTICLES	INFORMAL SAMPLES		FORMAL SAMPLES.
	Quantity.	Approximate Price.	If different Quantity.
Foods.			
Milk	4 oz.	1d.	1 pint, or $\frac{1}{2}$ pint from shop.
Dried milk	$\frac{1}{2}$ lb.	10d.	
Cream	1 pot.	4 $\frac{1}{2}$ d.	1 pot, 9d.
Butter	$\frac{1}{4}$ lb.	4d.	$\frac{1}{2}$ lb., if possible.
Margarine	$\frac{1}{4}$ lb.	2 $\frac{1}{2}$ d.	$\frac{1}{2}$ lb.
Lard	$\frac{1}{4}$ lb.	3 $\frac{1}{2}$ d.	$\frac{1}{2}$ lb.
Cheese	$\frac{1}{4}$ lb.	3d.	1 lb.
Tea	$\frac{1}{4}$ lb.	from 3d.	
Coffee	2 oz.	4d.	$\frac{1}{4}$ lb.
Chicory	$\frac{1}{4}$ lb.	3d.	$\frac{1}{2}$ lb.
Cocoa	$\frac{1}{4}$ lb.	5d. to 9d.	
Arrowroot	2 oz.	4d.	$\frac{1}{4}$ lb.
Rollod oats	$\frac{1}{2}$ lb.	2d.	1 lb.
Oatmeal, semolina, tapioca, rice, ground rice	$\frac{1}{2}$ lb.	2 $\frac{1}{2}$ d.	1 lb.
Pearl barley	$\frac{1}{2}$ lb.	3d.	1 lb.
Macaroni, vermicelli	$\frac{1}{2}$ lb.	4d.	
Cornflour	$\frac{1}{4}$ lb.	3d.	
Bread	small loaf	2 $\frac{1}{2}$ d.	
Flour	1 $\frac{1}{2}$ lb.	4 $\frac{1}{2}$ d.	
Sugar	$\frac{1}{2}$ lb.	2d.	1 lb.
Treacle	1 lb.	4 $\frac{1}{2}$ d.	
Golden syrup	1 lb.	5 $\frac{1}{2}$ d.	
Vinegar	—	1d.	1 pint.
Mustard	2 oz.	5d.	$\frac{1}{4}$ lb.
White pepper	1 oz.	3d.	$\frac{1}{4}$ lb.
Ground nutmeg, cinnamon, mace	1 oz.	6d.	
Baking, egg, custard, powder	1 packet	2d.	3 packets.
Sausage	$\frac{1}{2}$ lb.	6d.	1 lb.
Sultanas	$\frac{1}{2}$ lb.	4d. to 6d.	1 lb.
Beer	$\frac{1}{2}$ pint.	2 $\frac{1}{2}$ d.	1 quart.
Spirits	$\frac{1}{2}$ quartern.	1s. 5d. to 1s. 8d.	Quartern.
Drugs.			
Seidlitz powders	2	3d. or 4d.	1 doz.
Compound liquorice powder	2 oz.		
Boric acid powder	2 oz.	2d.	$\frac{1}{4}$ lb.
Pure borax	2 oz.		$\frac{1}{4}$ lb.
Bicarbonate of soda	2 oz.	2 $\frac{1}{2}$ d.	$\frac{1}{4}$ lb.
Cream of tartar, tartaric acid	1 oz.	3d.	2 oz.
Glauber salt	$\frac{1}{4}$ lb.	2d.	
Rochelle salt	$\frac{1}{4}$ lb.	2 $\frac{1}{2}$ d.	
Pure glycerin	1 oz.		
Olive oil, camphorated oil	1 oz.	3d.	2 oz.
Tinctures	1 oz.		3 or 4 oz.
Tablets	25		
Ointments	small tin.	2d.	1 large tin, or 2 small tins.

SPECIMEN PAGE OF SAMPLES BOOK

It would be better if "Food and Drugs (Adulteration) Act, 1928" were printed on each label. Size of page $6\frac{1}{4}$ inches by $3\frac{3}{4}$ inches (see p. 17).

Left-hand side.

Right-hand side.

CITY OF BIRMINGHAM HEALTH DEPARTMENT. Article asked for.—Butter.	A * No. 6576
CITY OF BIRMINGHAM HEALTH DEPARTMENT. Article asked for.—Butter.	A No. 6576
CITY OF BIRMINGHAM HEALTH DEPARTMENT. Article asked for.—Butter.	A No. 6576

CITY OF BIRMINGHAM No.
HEALTH DEPARTMENT. A
6576

Date.—Wednesday, Oct. 15, 1931.
Time.—11.10 a.m.
Article asked for.—Butter.
Price paid.—5d. per $\frac{1}{4}$ -lb.
Where purchased—
 Huckster's shop.
Vendor's Name and Address :—
 Mrs. Sally Butterwell,
 108, Bread Street,
 Bordesley Green.

Remarks.

Served by Miss Amelia Changit,
 in plain paper from an
 unlabelled piece about 4 lb.
 on table at back of shop.

Sampling Officer.—Watchem.

Agent.—Mrs. Keeneigh.

(Back of gummed labels.)

* One label would be on sample left with vendor ; the other two on samples taken away by the Sampling Officer.

REPORT AND EVIDENCE ON PURCHASE BY AGENT

WATCHEM *v.* BUTTERWELL (A 6576)

MRS. IVA KEENEIGH states as follows :—

On Wednesday, October 15th, 1931, at about 11.10 a.m., acting on the instructions of Sampling Officer Watchem, I went to a shop, No. 108 Bread Street, Bordesley Green, kept by Sally Butterwell, and asked for $\frac{1}{4}$ lb. butter which was served to me by a young woman. I paid 5*d.* for the article, which was handed to me wrapped in plain paper. It was cut from an unmarked piece about 4 lb. in weight, which was on a table at the back of the shop.

After I had been served Sampling Officer Watchem came into the shop, and I handed the purchase to him.

SAMPLING OFFICER ISHAL WATCHEM states as follows :—

On Wednesday, October 15th, 1931, at about 11.10 a.m., I sent Mrs. Iva Keeneigh to a huckster's shop, No. 108 Bread Street, Bordesley Green, for $\frac{1}{4}$ lb. butter. After she had been served, I went into the shop and complied with the Act.

Mrs. Keeneigh pointed out the young women who had served her, who gave her name as Amelia Changit. In her presence I asked Mrs. Keeneigh what she had asked for, where she was served from, and how much she paid for it. She said "Butter," and pointed to a substance about 4 lb. in weight, unlabelled, which was on a table at the back of the shop, and said she paid 5*d.* It was wrapped in plain paper, which I produce, and also the third sample.

CERTIFICATE A.6576

RESULT OF ANALYSIS :—

I am of opinion that the said sample was margarine.

Informal sample

CERTIFICATE A.6563.

Mrs. Keeneigh also went to this shop on October 10th, 1931, for $\frac{1}{4}$ lb. of butter. She paid 5*d.* for this $\frac{1}{4}$ lb., which was also wrapped in plain paper.

This sample, on analysis, was found to be margarine.

REPORT AND EVIDENCE OF MILK TAKEN IN COURSE OF DELIVERY

WATCHEM *v.* WATERS (A.1031)

SAMPLING OFFICER ISHAL WATCHEM states as follows :—

On Sunday, November 29th, 1931, I was in Milk Street outside the premises of Messrs. Sharp & Co. At 10.40 a.m. I saw a Ford motor lorry (XY.1234) drive up. The driver, Thomas Austin, of White Pump Cottage, Carter Lane, took off a churn of milk consigned by Mr. Will Waters, Farmer, of Waterworks Farm, Brookfields, Worcestershire, to Messrs. Sharp & Co.

After thoroughly mixing the milk I took a sample from the churn, which contained about 17 gallons. The churn and the lid were each numbered 23, and the lid and the side of the churn were each marked

"Sharp & Co., Birmingham." Attached to the lid was a guarantee label "Morning 17. Guaranteed new and pure milk with all its cream," which I now produce.

I then and there divided the sample into three parts, and sealed and labelled each part "A.1031." One part I submitted the same day to the City Analyst ; another I sent by registered parcel post with the letter of declaration (see p. 26), the same day addressed to Mr. W. Waters, Waterworks Farm, Brookfields, Worcestershire ; and I now produce the third sample.

CERTIFICATE A.1031.

RESULT OF ANALYSIS :—

Solids-not-fat 7·9 %. Fat 2·4 %.

The milk was deficient of 7 % of the minimum amount of solids-not-fat, and of 20 % of the minimum amount of fat, of which 7 % was due to the addition of water.

Notice to the Farmer

On Monday, November 30th, 1931, the Medical Officer of Health for the City wrote to Mr. Waters, informing him that a sample of milk consigned to Messrs. Sharp & Co., Milk Street, Birmingham, had been taken for analysis, and had been found not to comply with the limits laid down by the Ministry of Agriculture and Fisheries. The letter also offered to send a Sampling Officer to the farm to see the cows milked and to take a sample of the milk for analysis, to ascertain if the quality of the samples was due to the condition of the cows. A prepaid telegram was enclosed for a reply and particulars as to the number of cows and time of milking.

On December 1st a telegram was received from Mr. Waters accepting the offer, and stating that there were 17 cows and that they were milked at 4 p.m.

Visit to the Farm

Accordingly, on Tuesday, December 2nd, 1931, in company with Sampling Officer Lukhard, I visited the Waterworks Farm, Brookfield. Mr. Waters showed me the cooler, which was in good working order ; I asked about the food and was told the cows were fed on ground wheat and barley, wheat straw and hay. There were 26 cows in milk, all shorthorns, and in good condition, aged 3 to 8 years. I went into the dairy but did not see a separator.

The cows were milked by the farmer and his servant Isaac Pumpit, milking lasting from 4 to 5 p.m., and the 17 cows yielded about 16 gallons of milk. I was informed that the milking was done as usual, that the previous milking was at 6 a.m., and that the milk was left all night in an unlocked shed. I took a sample of the milk, and divided it into three parts, each being sealed and marked "A.1035," leaving one sample with the farmer.

CERTIFICATE A.1035.

RESULT OF ANALYSIS :—

Solids-not-fat 8·7 %. Fat 3·8 %.

The milk was genuine and of good quality.

A sample of the water at the farm was taken in my clean jug, and part of it submitted to the City Analyst (he reported the presence of nitrates in it).

Informal Sample

On the previous Sunday, November 22nd, about 10.50 a.m., I took an informal sample of milk from an unopened churn of milk sent by Mr. Waters to Messrs. Sharp & Co.

CERTIFICATE A.1020.**RESULT OF ANALYSIS :—**

Solids-not-fat 7·3 %. Fat 3·0 %

The milk was deficient of 14 % of minimum amount of solids-not-fat.

NOTE. The last two certificates relating to samples not taken under the conditions of the Act are not evidence, unless personally proved by the analyst.

**THIRD PARTY INFORMATION IN RELATION TO MILK TAKEN
IN COURSE OF DELIVERY**

SAMPLING OFFICER ISHAL WATCHEM states as follows :—

On Sunday, November 29th, 1931, I was in Milk Street outside the premises of Messrs. Sharp & Co. At 10.40 a.m. a Ford motor lorry (XY.1234), owned and driven by Thomas Austin, White Pump Cottage, Carter Lane, came up.

I said, "Have you any milk from Mr. Waters?" He said, "Yes, one churn, which Mr. Waters handed to me about 10 o'clock this morning." I asked him if he delivered it in the same state as he received it, and he said, "Yes."

There was a guarantee label on the churn, and I asked the driver to check it, and to watch me take a sample; which he did. I asked him when his next visit would be made to Mr. Waters' farm, and he said "To-morrow morning."

CERTIFICATE No. A.1031.**REPORT AND EVIDENCE OF PUBLIC ANALYST**

I now produce the certificate for sample of milk No. A.1031 (see p. 572), which I received from Sampling Officer Watchem on November 29th, 1931.

The sample of milk taken by him at the farm, A.1035, contained 8·7 % of solids-not-fat, and 3·8 % of fat, was genuine and of good quality.

The mixture of 15·5 gallons of farm milk with 1·5 gallons of water would give 17 gallons of "milk," containing 7·9 % of solids-not-fat as in sample A.1031. The fat, however, in such a mixture would be 3·5 %, and the removal of fat equivalent to about 2 lb. of butter would be necessary to reduce the fat to 2·4 %, as is present in the sample.

The freezing-point of sample A.1031 was -0.486°C ., which indicates the presence of 10 % added water, the freezing-point of the farm sample being -0.539°C .

A sample of water taken at the farm contained nitrates, which were also present in A.1031, but not in the farm sample.

During the month of November, 100 samples of milk, not sent from this farm, were analysed, the average composition of them was, solids-not-fat 8·75 % and fat 3·85 %; none of them contained less than 8·3 % of solids-not-fat, or less than 3·0 % of fat.

C. ANALYST'S ADULTERATED SAMPLES BOOK (Foolscap)

LEFT-HAND SIDE.

Article.	No.	Day.	Adulteration.	Fit ?

RIGHT-HAND SIDE.

Weight, Ounces.	Submitted by	Date of Certi- ficate.	Fines.			Costs.			Remarks.
			£	s.	d.	£	s.	d.	

D. ANALYST'S MISCELLANEOUS SAMPLES BOOK (Foolscap)

Day.	Articles.	From.	No.	DEPARTMENT.			
				H.	W.	P.W.	O.

NOTE. The Departments referred to above are :—Health, Water, Public Works, Others.

C. MILK PROSECUTION (Foolscap)

Town Arms, and
Public Analyst's
name.

CITY OF BIRMINGHAM

PUBLIC ANALYST'S CERTIFICATE

SAMPLE No. *A.1031*

To Mr. *Ishal Watchem*.

I, the undersigned, public analyst for the City of Birmingham, do hereby certify that on the *29th day of November, 1931*, I received for analysis from you a sample of MILK (numbered as above) which then weighed about $3\frac{1}{2}$ ounces. I have analysed the same, and declare the result of my analysis to be as follows :—

I am of opinion that the said sample contained the parts as under :—

Solids-not-fat	.	.	.	7.9
Fat	.	.	.	2.4
Water	.	.	.	89.7
				<hr/> 100.0

Comparison of the above results with the minimum limits fixed by the Sale of Milk Regulations, 1901, viz. : Solids-not-fat 8.5 per cent., Fat 3 per cent., shows that the said sample of Milk was deficient of *seven per cent. of the said minimum amount of solids-not-fat and of twenty per cent. of the said minimum of fat, of which seven per cent. was due to the addition of water.*

The freezing point was — 0.486° C., whereas the freezing point of milk taken at the farm was — 0.539° C., figures which indicate the presence of about *ten per cent. of added water.*

The sample also contained nitrates, indicating the presence of extraneous water.

OBSERVATIONS

No decomposition had taken place in the article that would interfere with the analysis.

As witness my hand this *1st day of December, 1931.*

.....
at Birmingham.

D. CAMPHORATED OIL PROSECUTION (Foolscap)

Town Arms, and
Public Analyst's
name.

CITY OF BIRMINGHAM

PUBLIC ANALYST'S CERTIFICATE

SAMPLE No. *B.991*To Mr. *Hiam Lukhard*.

I, the undersigned, public analyst for the City of Birmingham, do hereby certify that on the *29th day of February, 1931*, I received for analysis from you a sample of *Camphorated oil* (numbered as above), which then weighed about $2\frac{2}{3}$ ounce. I have analysed the same, and declare the result of my analysis to be as follows :—

I am of opinion that the said sample contained the parts as under, and the percentages of foreign ingredients as under.

<i>Camphor</i>	5
<i>Colza oil</i>	95
<hr/>					
<i>Total</i>	100

OBSERVATIONS

Camphorated oil should contain :—

<i>Camphor</i>	20
<i>Olive oil</i>	80
<hr/>					
<i>Total</i>	100

No change had taken place in the constitution of the article since purchase that would interfere with the analysis.

As witness my hand this *1st day of April, 1931*.

.....
at Birmingham.

FORMS OF SUMMONS

A. SUMMONS FOR MILK TAKEN IN COURSE OF DELIVERY

IN THE CITY OF BIRMINGHAM

To *Will Waters*, of *Waterworks Farm, Brookfields*, in the *County of Worcestershire*, *Farmer*.

INFORMATION has been laid this day by *Ishal Watchem*, a Sampling Officer for the said City of Birmingham, duly authorised in this behalf by the Local Authority (that is to say), the Council of the said City, for that on the *twenty-ninth* day of *November 1931*, at the City aforesaid, the said *Ishal Watchem* at the place of delivery, to wit, *Milk Street*, in the said City, procured a Sample of milk in course of delivery from you the said *Will Waters, Farmer*, to *Sharp and Company, Limited*, of *Milk Street*, in the said City, Milkmen, the purchasers or consignees thereof in pursuance of a contract for the sale to the said *Sharp and Company, Limited*, of such milk, and the said *Ishal Watchem*, suspecting the same to have been sold contrary to the provisions of Sections 2 and 16 of the Food and Drugs (Adulteration) Act, 1928, submitted the said sample to be analysed by the Public Analyst of the said City, and the said sample was analysed by the said Analyst, and was found by him to be not of the *quality* of the article contracted to be sold, the said Milk being deficient of solids, not fat, to the extent of *seven* per cent., and fat to the extent of *twenty* per cent. of the minimum amounts fixed by the Sale of Milk Regulations, 1901, and having been sold and consigned by you the said *Will Waters* to the prejudice of the purchasers contrary to the Statute in such case made and provided.

You are therefore hereby Summoned to appear before the Court of Summary Jurisdiction, sitting at the VICTORIA LAW COURTS, in Corporation Street, in the said City, on Friday, the *second* day of *January*, 1932, at the hour of Ten o'clock in the forenoon, to answer to the said information.

Dated this *tenth* day of *December*, One Thousand Nine Hundred and *Thirty-one*.

Justice of the Peace for the City aforesaid.

B. SUMMONS FOR CAMPHORATED OIL

IN THE CITY OF BIRMINGHAM

To *David Stores*, of *42 Rookery Street*, in the said City ; *Drysalter*.

INFORMATION has been laid this day by *Hiam Lukhard*, a Sampling Officer for the said City of Birmingham, duly authorised in this behalf by the Local Authority (that is to say), the Council of the said City, for that on the *twenty-ninth* day of *February* 1931, at the City aforesaid, you the said *David Stores*, did unlawfully sell by your Servant and Agent *Arthur Littleboy* to the prejudice of the said *Hiam Lukhard* the purchaser, a certain article of, Drug (to wit) *Camphorated Oil*, *the said Camphorated Oil being deficient of Camphor, and containing no Olive Oil*, which was not of the *quality* of the article demanded by such purchaser, contrary to Section 2 of the Food and Drugs (Adulteration) Act, 1928, and contrary to the Statute in such case made and provided.

You are therefore hereby summoned, etc.

C. BODY OF SUMMONS FOR RETAIL SALE OF UNMARKED MARGARINE

you being a person dealing in Margarine within the meaning of the Food and Drugs (Adulteration) Act, 1928, did unlawfully fail to conform to the regulation of the said Act (that is to say), that you did by your servant and agent *Amelia Changit* then and there while selling Margarine by retail deliver to the said *Ishal Watchem*, he being the purchaser of the said parcel, a certain parcel of Margarine without a paper wrapper on the outside of which was printed in capital block letters not less than half an inch long the word "**MARGARINE**," Contrary to the said Act.

You are therefore hereby summoned, etc.

D. BODY OF SUMMONS FOR EXPOSURE FOR SALE OF
UNMARKED MARGARINE

you being a person dealing in Margarine within the meaning of the Food and Drugs (Adulteration) Act 1928, did unlawfully fail to conform to the regulation of the said Act (that is to say), that you did then and there expose for sale by retail a certain parcel of Margarine there not being attached to such parcel in such a manner as to be clearly visible to the purchaser a label marked "MARGARINE" in printed capital letters not less than one and a half inches square Contrary to the said Act.

You are therefore hereby summoned, etc.

E. DECLARATION OF SERVICE ON BACK OF EACH SUMMONS

I, of the , Police Constable, hereby solemnly declare that I did on day the day of 193 , serve of with this Summons, by delivering a true copy thereof to him [or by leaving a true copy thereof with for him at being his (last or) most usual place of abode.]

Declared before me the }
day of 193 }

Justice of the Peace for the City aforesaid.

ADDENDA

Sampling, p. 17. In prosecution at Burnley, the difference between the added water in two samples (one had 6.2 %, the other 5.95 %) was attributed to one being put in a dry bottle and the other into a damp one (*B.F.J.*, 1932, 40).

Benzoic acid, p. 97. Mohler's test for benzoic acid and its determination have been studied by Illing (*Analyst*, 1932, 57, 224).

Milk, p. 200. See also "The Feeding of Dairy Cows," Ministry of Agriculture Bulletin. No. 42, 1932.

P. 215. *Evesham*. In 1932 the same cowman was fined £3 for stealing and damaging his master's milk.

P. 219. Comparative results for the amount of added water calculated from freezing points and percentages of solids-not-fat have been given by the Somerset County Analyst (*Analyst*, 1932, 57, 246).

Cream, p. 265. *London, Old Street*. Fat other than milk fat at least 30 %. The vendor was ordered to pay 5 guineas costs. A sample taken from his wholesale dealer was certified to contain at least 25 %, but the Government analysts found not less than 5 % of foreign fat. Fine £3 and 5 guineas costs (*Grocer*, 1932, May 7).

Cod liver oil, p. 274. For vitamin A, see "Report of the Permanent Commission on Biological Standardisation" (*Analyst*, 1932, 57, 173), also report (*Analyst*, 1932, 57, 302).

Linseed oil, p. 275. See also Cocchinaras, "The Composition of Linseed Oil" (*S.P.A.*, 1932, 57, 233).

Caviare, p. 291. This section was accidentally misplaced. *London, Marlborough Street*. Red caviare containing 6 grains per lb. of boric acid. Fine £3 (*Grocer*, 1932, April 23).

Sherry wine, p. 378. *London, Old Street*. On appeal to the Divisional Court (*Grant v. Harriman*), the magistrates were directed to convict the defendant, who was subsequently fined £2 and 5 guineas costs (*Grocer*, 1932, April 9, 23; *Analyst*, 1932, 57, 310).

Ground ginger, p. 420. *London, Thames*. A summons against the wholesaler for wilfully selling ground ginger containing a preservative was dismissed, as the magistrate thought before the wholesaler could be convicted the same process of taking samples and having them analysed must be repeated. An appeal to the High Court (*Twynham v. Badcock*) was allowed, and the magistrate was directed to convict (*Grocer*, 1932, April 9; *Analyst*, 1932, 57, 310).

Aspirin tablets, p. 519. *London, Old Street*. "Five grains" tablets which contained only 1 grain of aspirin, and having 65.5 % of sodium sulphate (*Grocer*, also *P.J.*, 1932, April 23).

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